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DEVELOPMENT OF CFA CARBONACEOUS
ROCKET NOZZLE MATERIALS

by

R. J. Lockhart, S. A. Bortz, and M. A. Schwartz

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Leo A. Burkardt, Project Manager

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FINAL REPORT

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FOREWORD

The research described in this report was conducted by IIT Research Institute on NASA Contract NAS3-12043. The work was administered by the NASA-Lewis Research Center, Chemical Rockets Procurement Section, with Leo A. Burkardt acting as Project Manager.

The studies presented cover the period May 20, 1969 to May 20, 1970. They were conducted by the Ceramics Division of IITRI under the management of M. A. Schwartz and S. A. Bortz, with R. J. Lockhart as Project Engineer.

DEVELOPMENT OF CFA CARBONACEOUS
ROCKET NOZZLE MATERIALS

ABSTRACT

The purpose of this program was to develop a materials system of filler, binder and additives for use as a low-cost molding material in ablative nozzles for the 260-in. solid rocket motor. A petroleum coke and carbon black filler was used to improve density by controlling particle size distribution. A furfuryl ester resin was used for its high carbon residue after pyrolysis. Staple carbon fibers were incorporated to improve the strength and crack resistance of molded bodies. Physical and mechanical properties were measured. In static firing tests of two subscale nozzles, this material compared favorably in erosion rate with several other ablative systems.

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SUMMARY

The purpose of this program was to develop improved carbonaceous composite formulations for use in ablative low-cost nozzles for the 260-inch solid-fueled rocket.

The first phase of the work was a material development study involving all the major categories of ingredients — carbon filler, resin binder, and additives. The fillers studied were petroleum coke, Ceylon graphite and carbon black. Single-phase fillers were found to produce weak, unsatisfactory bodies. Combination fillers were tested, and 10 parts fine-particle petroleum coke to 1 part carbon black was selected for its efficient compaction properties. The extremely fine particle carbon black filled most of the inter-particle space in the coarser petroleum coke. The resin binder, which had been developed by IITRI in earlier work, consisted of a furfuryl ester resin, pitch, furfuraldehyde and catalyst. This type resin was used for its high carbon residue after pyrolysis (57%). The binder composition was maintained constant, while its proportion in the carbonaceous mixture was varied over a wide range during the study, eventually becoming fixed at 35%. Additives tested included silicon carbide, titanium carbide, carbon fibers and rayon fibers. The carbide powders, which were selected for potential strengthening and erosion resistance, made minor improvements in some properties, but not sufficient to justify their high cost. The carbon fibers, prepared by coating continuous yarn with binder, curing with heat to remove tackiness and cutting into half-inch lengths, improved both mechanical properties and erosion resistance. During the thermal shock and erosion of the plasma torch test, the fibers resisted cracking and prevented the opening of any cracks that started. A content of 8% of the batch weight was found to be optimum. Rayon fibers were investigated briefly and found to be less effective than carbon but worthy of further consideration.

Bodies were fabricated by compacting the thoroughly-mixed ingredients in a die or form. Low pressures (10 to 60 lb/in², 0.069 to 0.41 MN/m²) and high pressures (500 to 2000 lb/in², 3.45 to 13.80 MN/m²) both produced sound bodies of good properties under suitable conditions. Heating of the dies to 212-300°F (100-150°C) during pressing was essential for high density. Extensive plasma torch testing showed that the highest possible density was not always desirable.

The second phase of the work was material characterization. A large billet was pressed and cured to a density of 1.30 g/cc. Specimens were machined from the billet, and several mechanical and physical properties were measured for future reference and nozzle design assistance:

- impact strength
- compressive strength
- tensile strength
- shear strength
- density
- porosity
- thermal expansion
- thermal conductivity

The third phase was the fabrication of two nozzle assemblies, each consisting of three pieces — entrance, throat and exit. The three billets for the first set were compacted to an average density close to that of the material characterization billet. The first nozzle was fired before the second was fabricated so that suggested improvements could be incorporated in the subsequent model. Although the first firing was considered successful, the second set of billets was compacted to a higher density (average 1.54 g/cc) for the purpose of lowering the erosion rate further.

Nozzle 1 was fired 48 seconds at an average chamber pressure of 384 lb/in^2 (2.64 MN/m^2). The erosion rate in the throat was 7.95 mils/sec ($202 \mu\text{m/sec}$), and there were several randomly located regions of much deeper than average erosion. Nozzle 2 was fired 39 seconds at an average chamber pressure of 515 lb/in^2 (3.55 MN/m^2). The erosion rate in the throat was lower at 6.13 mils/sec ($156 \mu\text{m/sec}$), and the appearance of the interior was much improved as a result of the use of the higher density material. Firings of several nozzles from other programs and made from different forms of carbonaceous materials by various investigators showed that the IITRI No. 2 nozzle ranked very high in terms of erosion resistance.

DEVELOPMENT OF CFA CARBONACEOUS ROCKET NOZZLE MATERIALS

I. INTRODUCTION

The objective of this program was to improve and test fire an existing carbonaceous material which appeared well qualified for an application as a rocket nozzle liner. Typical materials which have been evaluated for this purpose by various investigators are phenolic, epoxy-novolac or polyphenylene resins in combination with carbon or silica powder, carbon or silica cloth, asbestos fibers and canvas. These composites are relatively difficult to fabricate and sometimes tend to delaminate in use. A plastic mixture of carbon powder, resin binder and appropriate additives was developed at IITRI several years ago for high-temperature applications.¹⁻³ It was designated CFA (cold formed with additives) and was found to be suitable for molding into rocket nozzle components or trowelling onto charred surfaces for refurbishing a fired component for reuse.

The proper selection of fabrication materials is important in controlling the subsequent rate of loss from the nozzle walls during a rocket motor firing. It is essential to maintain reasonably closely the designed geometry of the nozzle throughout the firing, especially in the area of the throat. It is equally important that material be removed uniformly and in a predictable manner. Excessive non-uniform surface recession may lead to deterioration of propulsion performance, alteration of the characteristics required from the thermal protection system and even loss of structural integrity.

Ablation, or material loss from a nozzle wall, occurs primarily by four means: (1) corrosion, (2) erosion, (3) thermal degradation and (4) spalling. Corrosion is caused by chemical reactions between the nozzle liner and the combustion gases. It is the major mechanism that influences the performance of reinforced ablative-plastic composites. Investigators have found

that carbonaceous composites are suitable for solid-fuel rockets, which have a relatively low content of oxidizing components in the propellant exhaust. In contrast, oxide materials are recommended for the nozzles of liquid-fueled rockets, where the content of oxidizing vapor species is high. Erosion results from the shearing forces and abrasive particles in the high-velocity gas stream passing through the nozzle. It can be minimized by designing a contour that will permit laminar flow of gases over the nozzle surfaces. The thermal degradation of resin binders results in depolymerization and loss of volatiles or crosslinking and charring. Initially, the rate of weight loss is reduced by the cooling effect of the degradation reactions. Spalling is the loss of relatively large pieces of nozzle material from local thermal stresses. It is dependent upon the rate of heat transfer to the liner surface from the propellant gases and conduction away from the liner surface through the bulk material. Most substances are unable to conduct the heat away sufficiently rapidly, especially at the beginning of a firing, so cracks form and some pieces may break off. The use of a fibrous reinforcement is valuable in preventing cracking and spalling.

Resin-bonded carbon ranks high as an ablative material for re-entry vehicle surfaces or rocket engine components. The resin may be converted to carbon by pyrolyzing the body before use or it may be converted during use. The latter alternative is appropriate for a single-use nozzle insert, where loss of material by ablation is reduced by the heat absorbed to carbonize organic constituents. Relevant properties of this material are high heat absorption capacity, very good thermal shock resistance, high strength-density ratio and superior ablation characteristics. Certain properties may be varied within useful limits by controlling the processing steps and by the incorporation of selected additives.

The fabrication techniques are simple, and no special equipment is required for processing. A large structure (e.g., liner for a 260-inch (6.6 meter) diameter rocket nozzle) may be constructed from small building blocks pressed in a simple die and assembled with or without an adhesive. By adding a small excess of resin or solvent, the material may be prepared in a wetter form resembling a mortar, which can be trowelled onto a surface of the same material for repairing the body. Further dilution of the mixture to a pourable state will permit casting shapes in a die, which may subsequently be dried and cured.

A major objective inherent in the overall requirements for this research is that the resulting nozzle components be low cost in comparison with those prepared with presently used materials and processes. The bulk of the weight of ingredients consists of large-tonnage, low-cost commercial products, while the more expensive materials are used in small proportions. The cost of fabrication should be low, since it involves only weighing the ingredients, mixing, screening and pressing to the desired shape. The CFA material may be compacted into a billet and machined, or pressed to the final shape and finish machined to close tolerances. In some applications it may be feasible to use the as-pressed bodies without further processing.

II. MATERIAL DEVELOPMENT STUDIES

A. Basic Formulation

1. Materials

The CFA system consists of three categories of materials: (1) filler, (2) binder, and (3) special purpose additives. The filler is a carbon or graphite powder, and the binder is a thermosetting furfuryl ester resin with small proportions of coal tar pitch and furaldehyde. Additives studied on this program included fibers for reinforcement and two carbides for improved strength and erosion resistance.

a. Filler

Carbonaceous fillers investigated during this phase of the work included calcined petroleum coke, Ceylon graphite and carbon black. They provided several particle sizes and shapes with which to modify the density and properties of the bodies. Initially the fillers were used separately and fabrication difficulties were encountered. The very fine, uniform particle size of carbon black made the material difficult to densify, and finished samples were weak. The very high binder content required to coat all the fine particles implied a serious weight loss problem during high-temperature firing. The plate-shaped particles of Ceylon graphite tended to line up perpendicular to the direction of pressing and subsequently delaminate under stress. The equi-axed particles of petroleum coke used alone produced bodies with marginal strength and weight loss properties. However, the results observed here, as well as former work,^{4,5} showed that equi-axed or "spherical" particles provided the best overall properties.

Several bodies were fabricated using mixtures of two fillers, and the disadvantages of single fillers were found to be relieved to a significant extent. A properly selected auxiliary filler extends the range of particle sizes beyond that provided by most single fillers. The combination of petroleum coke with a

small proportion of carbon black provided the best overall properties, apparently because of its superior distribution of particle sizes. The importance of size distribution cannot be overemphasized. The filling of interstices among larger particles by smaller particles permits a body to be compacted to a much higher density than is possible for a single uniform size.

The distribution of particle sizes in three filler powders was determined by the Andreasen pipette technique. The plots of weight percent vs. particle size are presented in Figures 1 and 2. The batch of petroleum coke used in the early part of the work was a very fine powder, more than 90% of it being smaller than 325 mesh ($44\mu\text{m}$). Twenty percent of the material was smaller than $2\mu\text{m}$, where the size distribution was unknown. When this coke was exhausted, a second batch was obtained from the same supplier. While it met the same overall sieve requirement ($> 90\%$ through 325 mesh), the percentage of each size smaller than $20\mu\text{m}$ was much lower than in the original powder. The lower proportion of fines necessitated a decrease in the binder content of the CFA mixture. This illustrates the importance of a thorough characterization of the materials used in a process. Each batch may be somewhat different, even when obtained from the same source.

The Ceylon graphite filler (Figure 2) covered nearly the same overall size range, but the percentages were concentrated in the upper part of the range. Only 3% of the material was smaller than $2\mu\text{m}$, while 56% was larger than $44\mu\text{m}$.

b. Binder

The binder is a mixture of a furfuryl ester resin developed by IITRI, coal tar pitch, furfuraldehyde as a solvent for the pitch and a hardening agent for the resin. The ester type resin has significant advantages over other resins, especially the conventional furfuryl alcohols. No solvents are used in its preparation, which can volatilize during thermosetting and leave an undesirable degree of porosity. In contrast to the alcohol, the

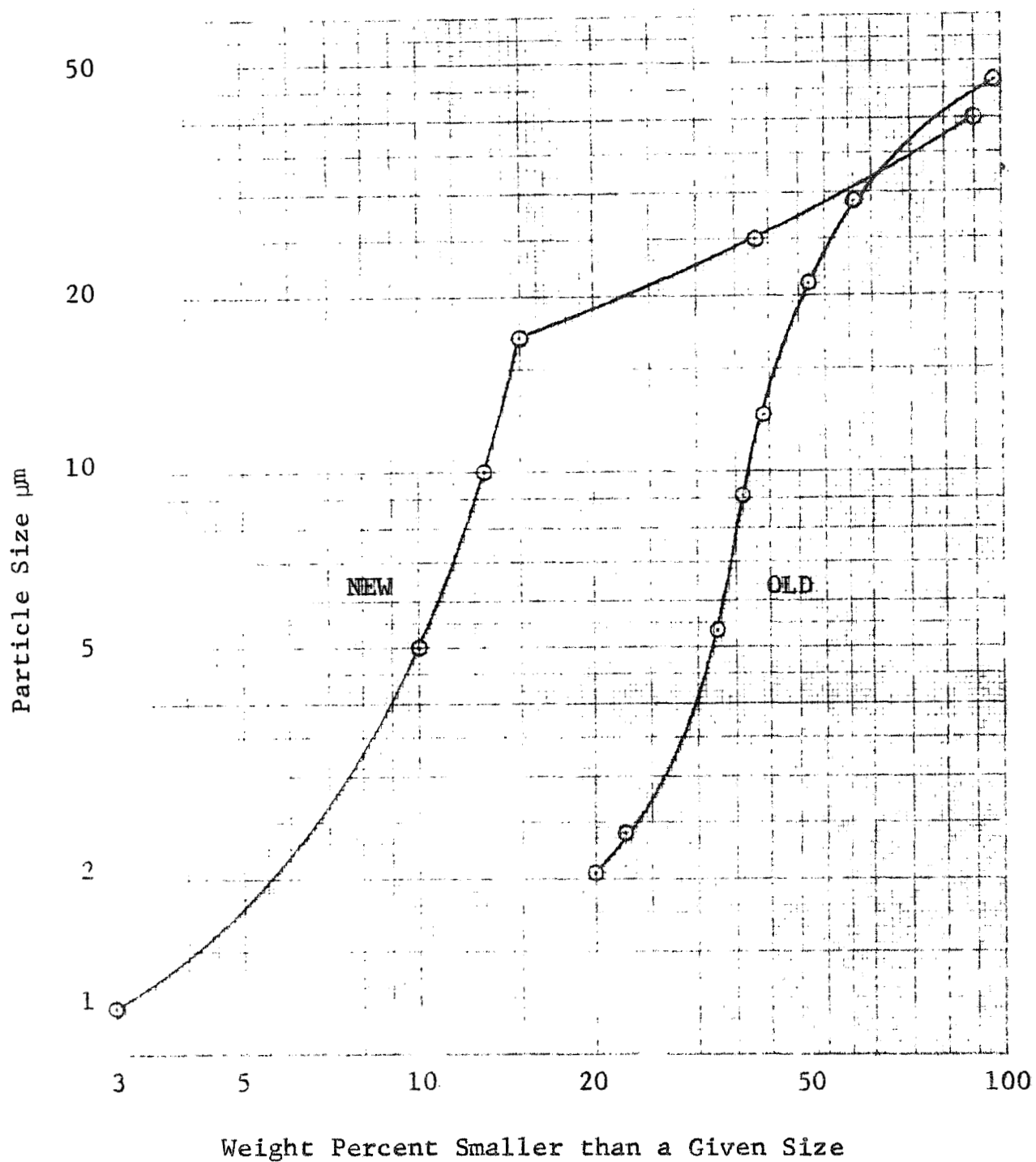


Fig. 1 PARTICLE SIZE DISTRIBUTION IN OLD AND NEW PETROLEUM COKE FILLER POWDERS.

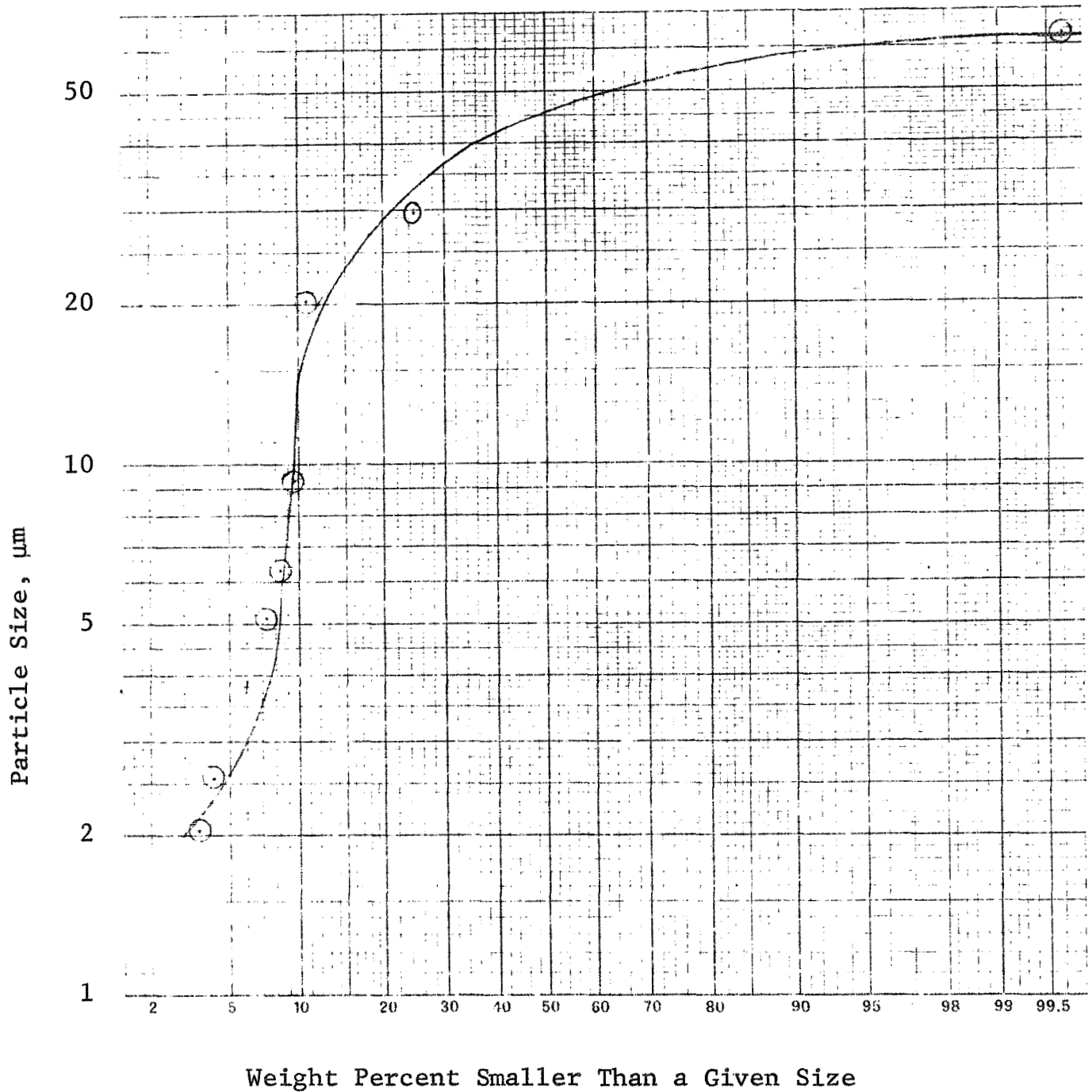


Fig. 2 PARTICLE SIZE DISTRIBUTION IN CEYLON GRAPHITE FILLER POWDER

ester polymerizes by addition and cross linking, so no condensation product is formed to cause trouble. During curing only about 1% of the starting weight of the resin is lost by volatilization. Studies by gas chromatography have shown that only low molecular weight gases are formed during pyrolysis. The carbon residue is relatively high (56-58%). See Table I for comparative data.

The composition of the binder (shown in Table II) remained constant throughout this work, while the proportion of binder in a CFA mixture was changed as required. The lower limit of the quantity used was determined by the requirement for the liquid to coat nearly all of the particles of filler and additives. Failure to coat a significant percentage of the solid particles resulted in a serious deterioration of strength and density in a sample fabricated from such a mixture. The upper limit for binder content was set by increasing difficulty of processing the gummy material. An additional limiting factor was the loss of weight during a plasma torch test, which was directly proportional to the amount of binder present in the body. The range of concentrations covered in this work was 30 to 55% of the batch weight.

The catalyst or hardening agent used was a saturated aqueous solution of zinc chloride. Previous experience with an active acid catalyst showed the disadvantages of using a substance which began to react as soon as it was mixed with the resin. Extended processing operations could not be completed before the mixture became excessively viscous. The zinc chloride solution is a latent catalyst which caused no apparent hardening during several days of standing at room temperature. When heated above 122°F (50°C), it began to react at a significant rate.

c. Additives

The CFA materials system is ideally suited to the incorporation of many types of solid or liquid additives. Carbides of silicon, titanium and other substances will aid in forming hard, strong bodies with high erosion resistance in a rocket

Table I
CARBON RESIDUE OF PYROLYZED RESINS (6)

Resin	Residue %
Furane	55
Phenolic	49
Silicone	43
Modified Furane	23
Melamine	9
Epoxy	4

Table II
BINDER COMPOSITION

Ingredient	Percentage
Furfuryl ester resin	70
Coal tar pitch	20
2-Furaldehyde	10
Catalyst	(Typically 4)

engine environment. Carbon or graphite in the form of yarn, staple fibers or cloth may be added to increase strength and to maintain the structural integrity of a stressed body. Of the many types of potentially useful additives, the ones investigated on this program were carbon yarn, silicon carbide and titanium carbide.

The carbon yarn used was a commercial product prepared from rayon and initially in the form of 1/4-in. (0.63 cm) staple fibers. After some work it was decided that these fluffy fibers might not be wetted sufficiently by the small proportion of binder present to promote good bonding with the matrix. Subsequently, continuous yarn was coated with binder, partially cured to permit easy handling and cut into short lengths. When pressed, each length of yarn contained its own supply of binder which softened in the heated die and bonded to the adjacent matrix.

The carbon yarn was selected because its mechanical properties were sufficient for the intended application. The superior characteristics of the graphite yarns were not considered necessary, especially when the unfavorable cost ratio of 8:1 was taken into account. Since the bulk of the cost is incurred in processing rayon into carbon, it was decided to try incorporating rayon yarn into a sample and converting it into carbon in situ during a plasma torch test. The cost advantage would be substantial, if the rayon reinforcement served its intended purpose. A description of both types of yarn is presented in Table III.

2. Fabrication Techniques

a. Mixing

The procedure for mixing the ingredients was developed for the purpose of obtaining intimate blending and a relatively small range of agglomerate sizes for efficient compaction. The solid constituents were mixed together in a twin-shell blender, and the liquids were stirred in a small egg-beater-type mixer. All constituents were then combined in a planetary blender with

Table III
PROPERTIES OF REINFORCING YARNS

	Carbon	Rayon
Type	VYB 70-1/2 ⁽¹⁾	5952 ⁽²⁾
Denier	1430	2200
Filaments	1440	1440
Filament diameter, μm	9.5	14.5
Twist, turns/inch	2.4S	2.4Z
turns/cm	0.94S	0.94Z

(1) Union Carbide Corporation

(2) American Enka Corporation

paddle attachment. The catalyst was a saturated water solution which was diluted with an equal weight of water to prevent the heat of mixing from starting the curing reaction. At the completion of mixing, the agglomerates were too damp for screening, so they were dried for a few minutes in an oven. Following this treatment they could be screened with relative ease. Staple fibers (when used) were added to the screened powder. The three techniques for forming bodies from the CFA powder are described in the following sections.

b. Casting

The method of forming specific shapes from CFA formulations consisted of three simple steps: (1) mix the ingredients, (2) compact in a die or form and (3) cure the resin. Early in the program a modest effort was devoted to preparing a liquid formulation which could be poured into a mold, dried and cured. Difficulties with thixotropy, drying cracks and excessive shrinkage could not be overcome in the limited time available. The remainder of the effort was spent on compaction in two pressure ranges, designated as "low" (10 to 60 lb/in², 0.069 to 0.41 MN/m²), and "high" (500 to 2000 lb/in², 3.45 to 13.80 MN/m²).

c. Low-Pressure Compaction

Low-pressure compacts were pressed in a cold die, then cured in an oven with no applied pressure. Initially, the compacting pressure was 10 lb/in² (0.069 MN/m²), but the resulting mechanical properties were inferior. When the pressure was increased to 20 lb/in² (0.137 MN/m²), the bodies were sufficiently dense and strong for the application. During curing, the samples tended to expand slightly, and the density decreased. This effect was less pronounced with formulations which compacted well and with those containing no fibers. Towards the end of the program, some relatively large samples were prepared at pressures as low as 13 lb/in² (0.090 MN/m²) with heat lamps irradiating the die while it was under load. The resin was partly cured, so the samples were hard and quite dense upon removal from the die.

d. High-Pressure Compaction

High-pressure compacts were prepared in a heated die on a semi-automatic press. The sample size was limited to 4-in. x 4-in. x 1-in. (10.2cm x 10.2cm x 2.5cm), from which specimens were cut for mechanical property tests. The pressing conditions were normally 500, 1000 or 2000 lb/in² (3.45, 6.89, or 13.80 MN/m²) 302°F (150°C) and a time of one hour under load to cure the resin thoroughly. The press automatically ejected each sample at the completion of the preset cycle.

B. Property Tests for Screening

Three screening tests were selected to evaluate the results of composition and processing changes in the CFA material: (1) density and porosity, (2) flexural strength and (3) erosion by a plasma torch.

1. Test Procedures

The density was determined by boiling the specimens in water for an hour and soaking them overnight. They were weighed suspended in water, in air while still saturated with water and in air after thorough drying in an oven. From these three weighings, the bulk density, apparent density and apparent porosity were calculated by the mathematical formulas in ASTM Standard C373-56.

Flexural strength was measured by 4-point loading of specimens about 3 in. (7.6cm) long with a 1/2 in. (1.27cm) square cross section. The span of the supporting edges was 2-1/2-in. (6.35cm) and the span of the loading edges was 1 in. (2.54cm). A deflectometer was mounted under the center of each specimen so that the modulus of elasticity could be calculated. The load vs. deflection curves were recorded on a strip chart.

An indication of the behavior expected of these materials during an actual rocket engine firing was obtained by placing small specimens in a high thermal flux environment. Cubes approximately 1 in. (2.54cm) on a side were exposed for 30 or

120 seconds to the flame of an arc plasma torch designed to produce a temperature range of 5600 to 5800°F (3093 to 3204°C) at the surface of the specimen. The cubes were mounted at a 45° angle to the flame, with the tip of the flame just touching the sample. The specimen surface temperature was measured with an optical pyrometer.

The data and calculations for the material-development formulations are summarized in Tables IV through VIII. These values were subsequently used in determining an optimum composition. The results are categorized into formulations with and without additives and bodies fabricated with high and low pressures. Few data are presented for single-component fillers, since they were discovered early in the program to be unsatisfactory. The samples produced from carbon black or Ceylon graphite were so poor that it would have been pointless to measure their properties.

2. Test Results

a. Density and Porosity

All of the screening properties were affected by the bulk density of the material. The highest density attained during the program was 1.60 g/cc, which is about 73% of the theoretical maximum. The highest values (1.50 to 1.60 g/cc) were generally obtained by the high-pressure (molding) process, because the resin was cured under heat and pressure, and no expansion could occur after the pressure was removed. In the low-pressure process, where the material was compacted at room temperature and subsequently cured without an applied load, the best density attained was 1.45 g/cc. The plasma torch evaluations showed that specimens of highest density cracked severely on the heated surfaces from thermal shock and probably also from inability of volatiles from the charring binder to escape rapidly through the relatively small number of open pores. The porosity of properly molded bodies ranged from 2 to 5%. In contrast, the samples compacted at low pressure, with densities of 1.20 to 1.40 g/cc and

Table IV

PROPERTIES OF HIGH-PRESSURE (MOLDED) CFA SAMPLES WITHOUT FIBERS

Sample	Compaction Pressure ⁽¹⁾		Binder %	Filler ⁽²⁾		Bulk Density g/cc	Apparent Density g/cc
	lb/in ²	MN/m ²		PC %	CB %		
19473-13-1	500	3.45	40	60	0	1.60	1.68
19473-13-2	1000	6.89	40	60	0	1.59	1.68
19473-13-3	2000	13.79	40	60	0	1.58	1.68
19473-6-1	500	3.45	45	44	11	1.54	1.62
19473-6-2	1000	6.89	45	44	11	1.57	1.62
19473-6-3	2000	13.79	45	44	11	1.55	1.62
19546-8-1	500	3.45	35	52	13	1.41	1.69
19546-8-2	1000	6.89	35	52	13	1.49	1.64
19546-8-3	2000	13.79	35	52	13	1.52	1.64

Table IV (Cont'd.)

PROPERTIES OF HIGH-PRESSURE (MOLDED) CFA SAMPLES WITHOUT FIBERS

Sample	Apparent Porosity %	Flexural Strength ⁽¹⁾		Modulus of Elasticity ⁽¹⁾		Plasma Torch Test ⁽³⁾ Weight Loss-%	
		lb/in ²	MN/m ²	lb/in ²	MN/m ²	30 sec.	120 sec.
19473-13-1	5.05	2690	18.55	916,000	6316	12.10	26.92
19473-13-2	5.06	2745	18.93	922,000	6357	—	—
19473-13-3	5.57	2788	19.22	861,000	5936	11.87	26.47
19473-6-1	4.58	3795	26.17	821,000	5661	14.17	26.28
19473-6-2	3.03	3571	24.62	879,000	6061	14.42	28.75
19473-6-3	3.18	3692	25.46	833,000	5743	15.38	28.91
19546-8-1	16.66	1674	11.54	898,000	6192	14.04	26.47
19546-8-2	9.27	2545	17.55	972,000	6702	13.82	26.30
19546-8-3	7.07	3478	23.98	1,137,000	7839	13.25	26.43

(1) N/m^2 (Newtons per square meter) = Kg/m^2 (9.806 m/sec^2 , standard acceleration of gravity).

(2) PC = petroleum coke, CB = carbon black

(3) The plasma torch test was conducted on different specimens from the same batch as those listed in the first column.

Table V
PROPERTIES OF HIGH-PRESSURE (MOLDED) CFA SAMPLES WITH FIBERS

Sample	Compaction Pressure ⁽¹⁾		Binder %	Filler ⁽²⁾		Fiber Content %	Bulk Density g/cc	Apparent Density g/cc	Apparent Porosity %
	lb/in ²	MN/m ²		PC %	CB %				
19473-13-2	1000	6.89	40	60	0	0	1.59	1.68	5.06
19562-20-1	1000	6.89	40	50	5	5	1.51	1.65	8.40
19562-20-2	1000	6.89	39	47	4	10	1.49	1.64	9.04
19553-20-3	1000	6.89	40	41	4	15	1.48	1.63	11.84
19553-20-4	1000	6.89	39	38	3	20	1.42	1.62	12.42
19758-4-1	5000	34.47	41	54	5	0	1.59	1.65	3.65
19758-4-2	5000	34.47	40	50	5	5	1.57	1.65	4.58
19758-4-3	5000	34.47	38	48	4	10	1.56	1.64	5.14
19758-4-4	5000	34.47	37	44	4	15	1.56	1.64	4.64
19758-4-5	5000	34.47	35	41	4	20	1.54	1.62	5.20

Sample	Flexural Strength ⁽¹⁾		Modulus of Elasticity ⁽¹⁾		Plasma Torch Test Weight Loss - %	
	lb/in ²	MN/m ²	lb/in ²	MN/m ²	30 sec.	120 sec.
19473-13-2	2745	18.93	922,000	6357	12.08	26.78
19562-20-1	2160	14.89	559,000	3854	13.16	27.96
19562-20-2	2870	19.79	708,000	4882	12.90	27.96
19553-20-3	3737	25.77	872,000	6012	12.67	28.54
19553-20-4	4832	33.32	792,000	5461	13.92	29.25
19758-4-1	3429	23.64	1,160,000	7998	-	-
19758-4-2	2867	19.77	1,089,000	7508	-	-
19758-4-3	3535	24.37	1,170,000	8067	-	-
19758-4-4	4454	30.71	1,253,000	8639	-	-
19758-4-5	3889	26.81	1,040,000	7171	-	-

(1) $\text{N/m}^2 = (\text{kg/m}^2)(9.806 \text{ m/sec}^2)$

(2) PC = petroleum coke, CB = carbon black

Table VI

PROPERTIES OF LOW-PRESSURE CFA SAMPLES WITHOUT FIBERS

Sample	Compaction Pressure ⁽¹⁾		Binder %	Filler ⁽²⁾		Bulk Density g/cc	Apparent Density g/cc	Apparent Porosity %
	lb/in ²	MN/m ²		PC %	CB %			
19319-5-1	10	0.069	35	65	0	1.17	1.75	33.29
19459-5-1	20	0.137	40	60	0	1.34	1.70	20.87
19473-14-1	20	0.137	35	52	13	1.28	1.72	25.30
19473-8-2	20	0.137	40	48	12	1.40	1.68	16.42
19546-18-1	20	0.137	40	54	6	1.45	1.67	13.12
19562-24-1	20	0.137	40	56	4	1.24	1.70	27.01
19758-23-1	20	0.137	35	60	5	-	-	-

Sample	Flexural Strength ⁽¹⁾		Modulus of Elasticity ⁽¹⁾		Plasma Torch Test Weight Loss - %	
	lb/in ²	MN/m ²	lb/in ²	MN/m ²	30 sec.	120 sec.
19319-5-1	412	2.84	84,000	579	6.4	21.4
19459-5-1	1326	9.14	307,000	2117	11.69	26.68
19473-14-1	808	5.57	212,000	1462	10.21	25.33
19473-8-2	2042	14.08	414,000	2854	13.24	23.73
19546-18-1	2466	17.00	827,000	5702	14.77	27.24
19562-24-1	1118	7.71	532,000	3668	-	-
19758-23-1	-	-	-	-	15.92	32.16

(1) $\text{NM/m}^2 = (\text{kg/m}^2)(9.806 \text{ m/sec}^2)$

(2) PC = petroleum coke, CB = carbon black.

Table VII
PROPERTIES OF LOW-PRESSURE CFA SAMPLES WITH FIBERS

Sample	Compaction Pressure ⁽¹⁾		Binder %	Filler ⁽²⁾		Fiber Content ⁽³⁾ %	Bulk Density g/cc	Apparent Density g/cc
	lb/in ²	MN/m ²		PC %	CB %			
19553-10-1	20	0.137	40	49	6	5	1.42	1.66
19553-5-1	20	0.137	40	45	5	10	1.38	1.66
19553-12-1	20	0.137	40	41	4	15	1.24	1.65
19562-19-1 ⁽⁴⁾	20	0.137	30	36	4	10	1.21	1.86
19758-24-1	32	0.220	33	52	5	10	1.38	1.70

Sample	Apparent Porosity %	Flexural Strength ⁽¹⁾		Modulus of Elasticity ⁽¹⁾		Plasma Torch Test Weight Loss - %	
		lb/in ²	MN/m ²	lb/in ²	MN/m ²	30 sec	120 sec
19553-10-1	14.17	2332	16.08	672,000	4633	13.62	26.93
19553-5-1	16.62	2487	17.15	635,000	4378	13.00	27.50
19553-12-1	26.93	2087	14.39	278,000	1917	14.59	30.02
19562-19-1 ⁽⁴⁾	35.02	779	5.37	254,000	1751	14.94	34.16
19758-24-1	18.82	-	-	-	-	20.63	37.98

(1) $\text{N/m}^2 = (\text{kg/m}^2)(9.806 \text{ m/sec}^2)$.

(2) PC = petroleum coke, CB = carbon black.

(3) All samples contained precoated carbon yarn, except the last which contained precoated rayon yarn.

(4) Contains 20% silicon carbide.

Table VIII

COMPARISON OF HIGH-PRESSURE SAMPLES CONTAINING 20% CARBIDE POWDER

Sample	Compaction Pressure ⁽¹⁾		Binder %	Filler ⁽²⁾		Additive	Bulk Density g/cc	Apparent Density g/cc
	lb/in ²	MN/m ²		PC %	CB %			
19473-13-1	500	3.45	40	60	0	None	1.60	1.68
19473-13-2	1000	6.89	40	60	0	None	1.59	1.68
19473-13-3	2000	13.79	40	60	0	None	1.58	1.68
19546-1-1	500	3.45	30	40	10	SiC	1.51	1.84
19546-1-2	1000	6.89	30	40	10	SiC	1.57	1.83
19546-1-3	2000	13.79	30	40	10	SiC	1.64	1.80
19459-15-8	500	3.45	30	50	0	TiC	1.78	1.96
19459-15-3	1000	6.89	30	50	0	TiC	1.90	1.96
19459-15-2	2000	13.79	30	50	0	TiC	1.92	1.96

Sample	Apparent Porosity %	Flexural Strength ⁽¹⁾		Modulus of Elasticity ⁽¹⁾		Plasma Torch Test Weight Loss - %	
		lb/in ²	MN/m ²	lb/in ²	MN/m ²	30 sec	120 sec
19473-13-1	5.05	2690	18.55	916,000	6316	12.10	26.92
19473-13-2	5.06	2745	18.93	922,000	6357	-	-
19473-13-3	5.57	2788	19.22	861,000	5936	11.87	26.47
19546-1-1	17.51	2728	18.81	820,000	5654	10.85	20.58
19546-1-2	13.88	3372	23.25	989,000	6819	10.80	20.12
19546-1-3	9.02	4256	29.34	1,234,000	8508	10.13	20.80
19459-15-8	8.97	1715	11.82	688,000	4744	13.01	27.19
19459-15-3	3.11	5338	36.80	1,361,000	9384	13.24	27.54
19459-15-2	2.20	3086	21.28	668,000	4606	13.44	28.04

(1) $\text{N/m}^2 = (\text{kg/m}^2)(9.806 \text{ m/sec}^2)$

(2) PC = petroleum coke, CB = carbon black.

porosities of 13 to 35%, showed both fewer and smaller cracks after plasma torch testing. Therefore it was essential to control the density to produce the desired results.

The addition of staple fibers to a CFA formulation presented a fabrication problem which was directly proportional to the fiber content. More reinforcement was always desirable to obtain the potential benefits of a fiber-matrix system, but as it was added, it became increasingly difficult to prepare a structure free of major flaws at the locations of undispersed fiber clumps. Thus a compromise was required to obtain the best combination of properties.

The effect of fiber content on CFA bodies is illustrated in Figures 3 through 9. The density decreases as more fibers are incorporated, the effect becoming more pronounced the lower the compacting pressure. The curves for porosity are almost the exact inverse of those for density. Both sets of curves show that there would be little benefit from a further increase in pressure, since the density change with fiber content becomes very small at higher pressures.

b. Flexural Strength and Modulus

The flexural strength and modulus of elasticity of unreinforced bodies were approximately proportional to the density, the higher values being measured, as expected, on the materials compacted at high pressures. Another factor which influenced the flexural properties was the content of carbon black in the filler. The particle size of this powder was 0.041 μ m (according to the manufacturer's literature), compared with a range of 1 to 46 μ m in the petroleum coke. Thus the carbon black could easily fill in the interstices in the coke to make a denser mixed filler. Test results showed that the flexural and other properties were optimum when the carbon black content was 4 to 6% of the batch weight.

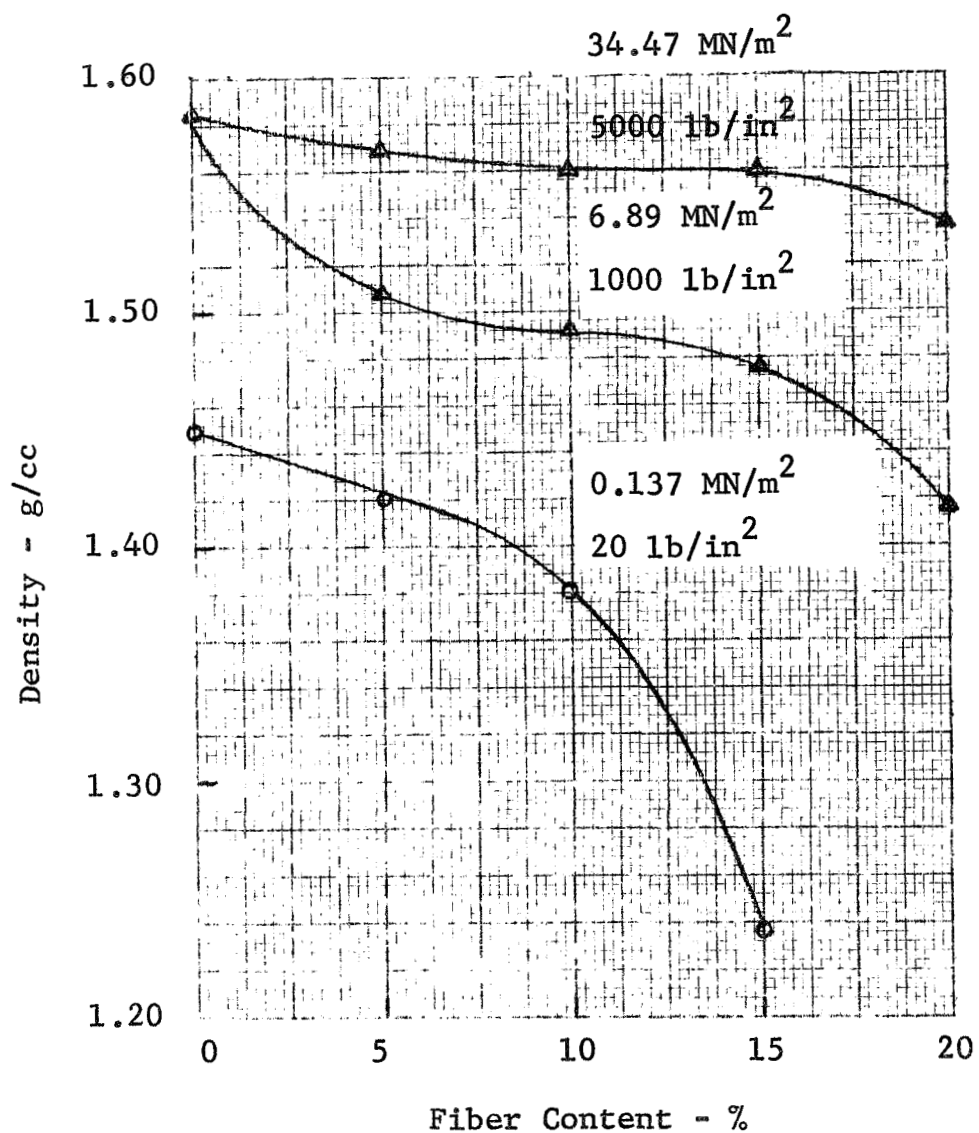


Fig. 3 EFFECT OF FIBER CONTENT ON DENSITY FOR CFA SAMPLES COMPACTED AT DIFFERENT PRESSURES.

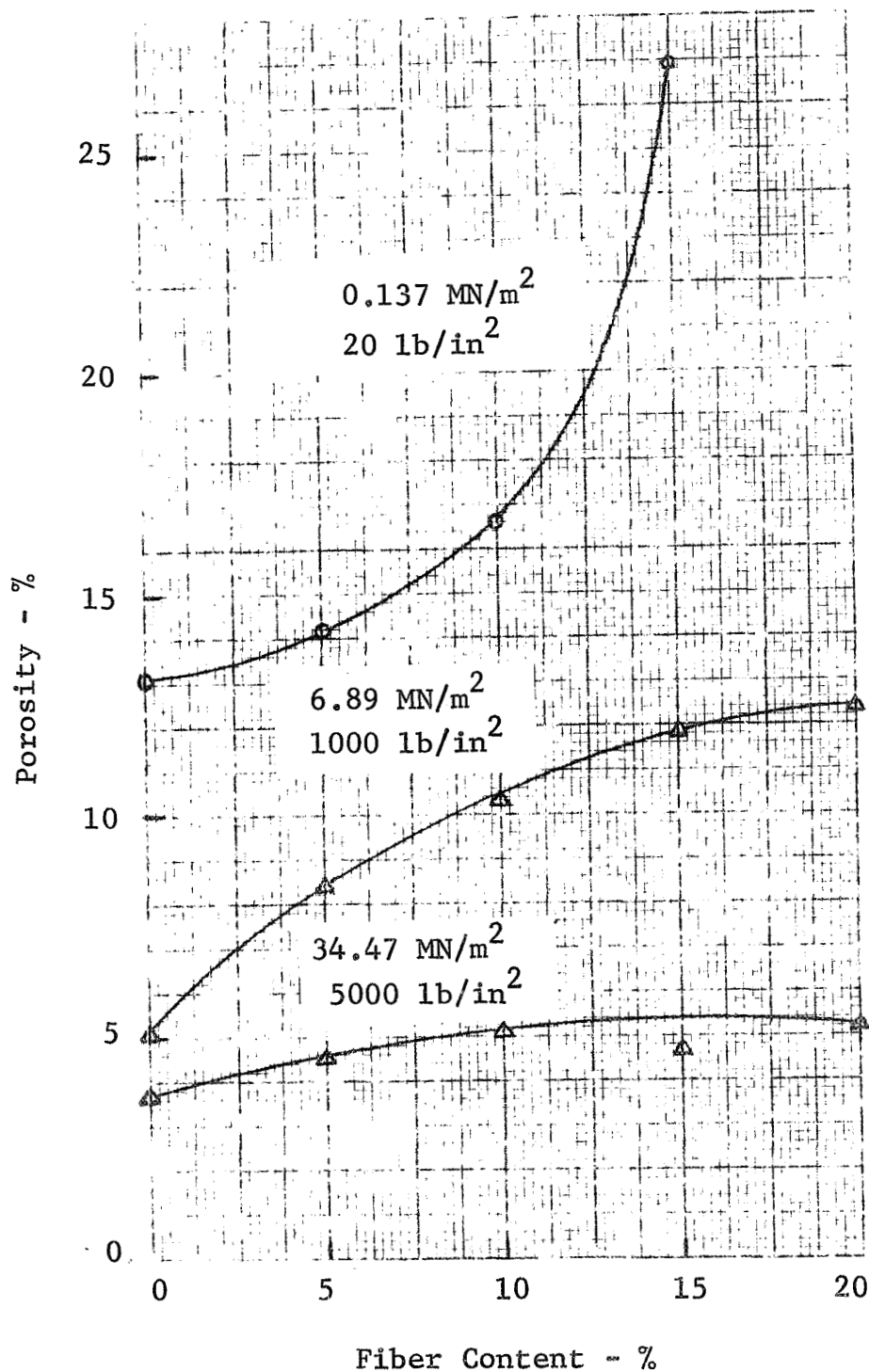


Fig. 4 EFFECT OF FIBER CONTENT ON POROSITY FOR CFA SAMPLES COMPACTED AT DIFFERENT PRESSURES.

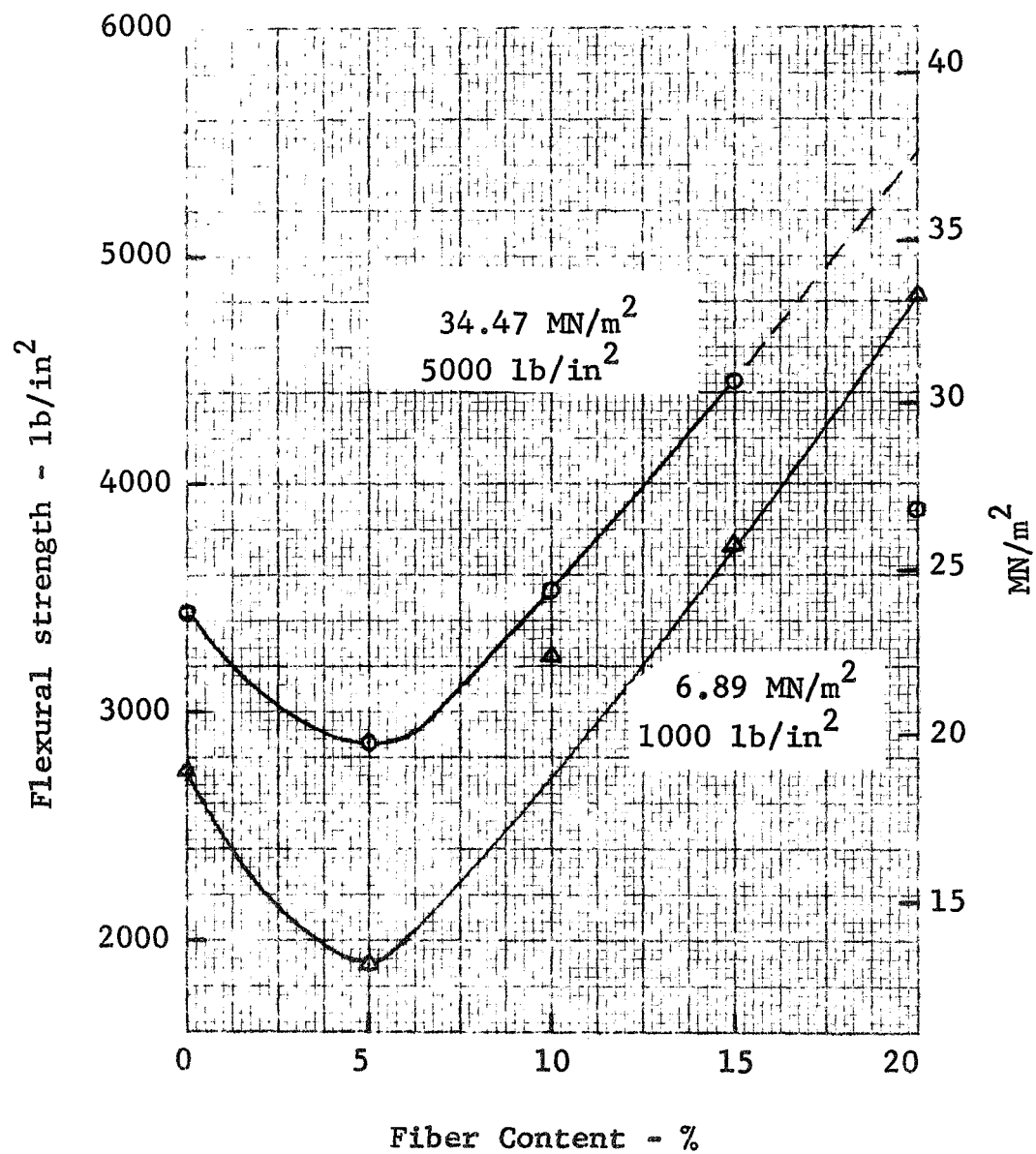


Fig. 5 EFFECT OF FIBER CONTENT ON FLEXURAL STRENGTH FOR CFA SAMPLES COMPACTED AT DIFFERENT PRESSURES.

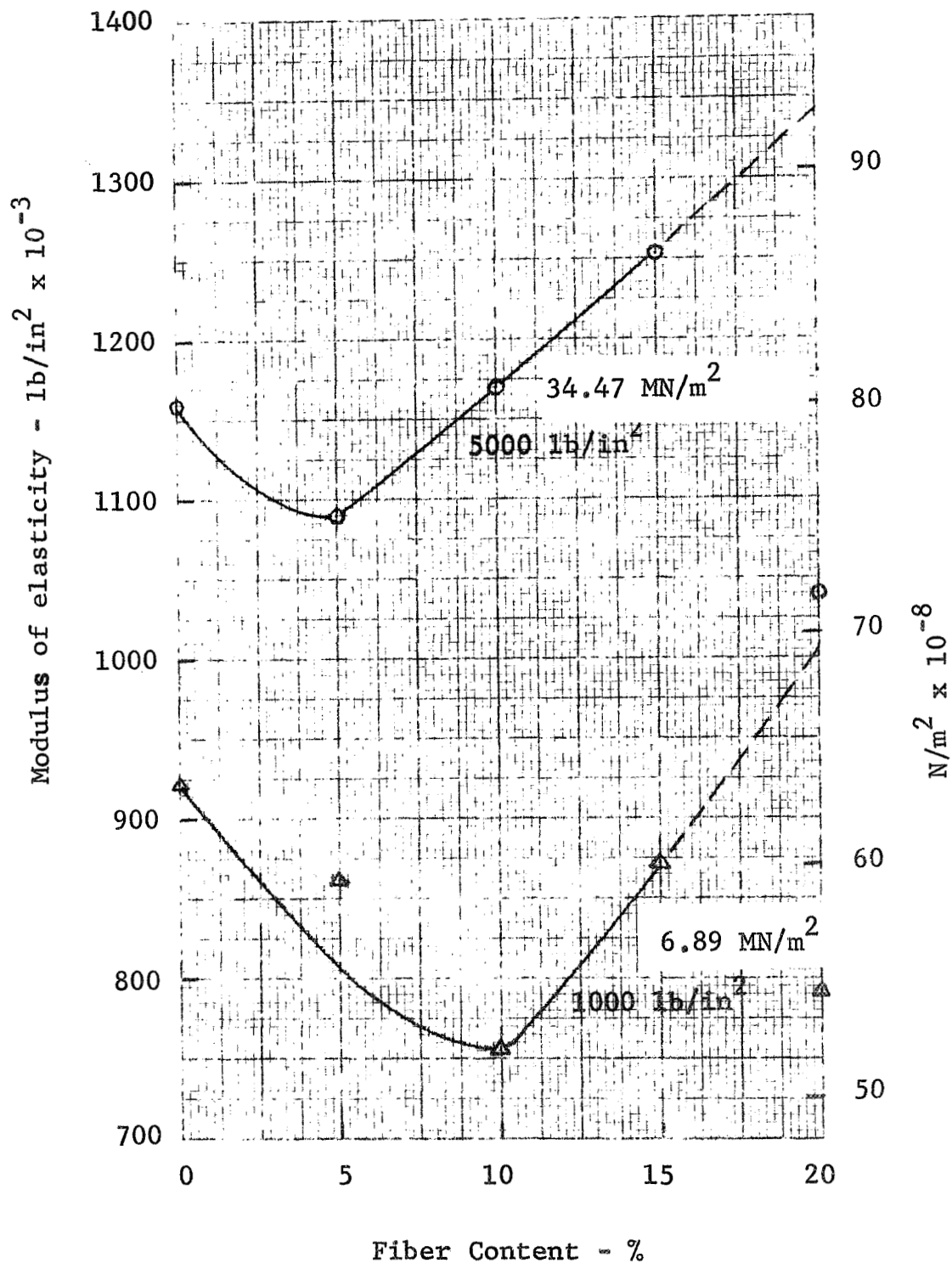


Fig. 6 EFFECT OF FIBER CONTENT ON MODULUS OF ELASTICITY FOR CFA SAMPLES COMPACTED AT DIFFERENT PRESSURES.

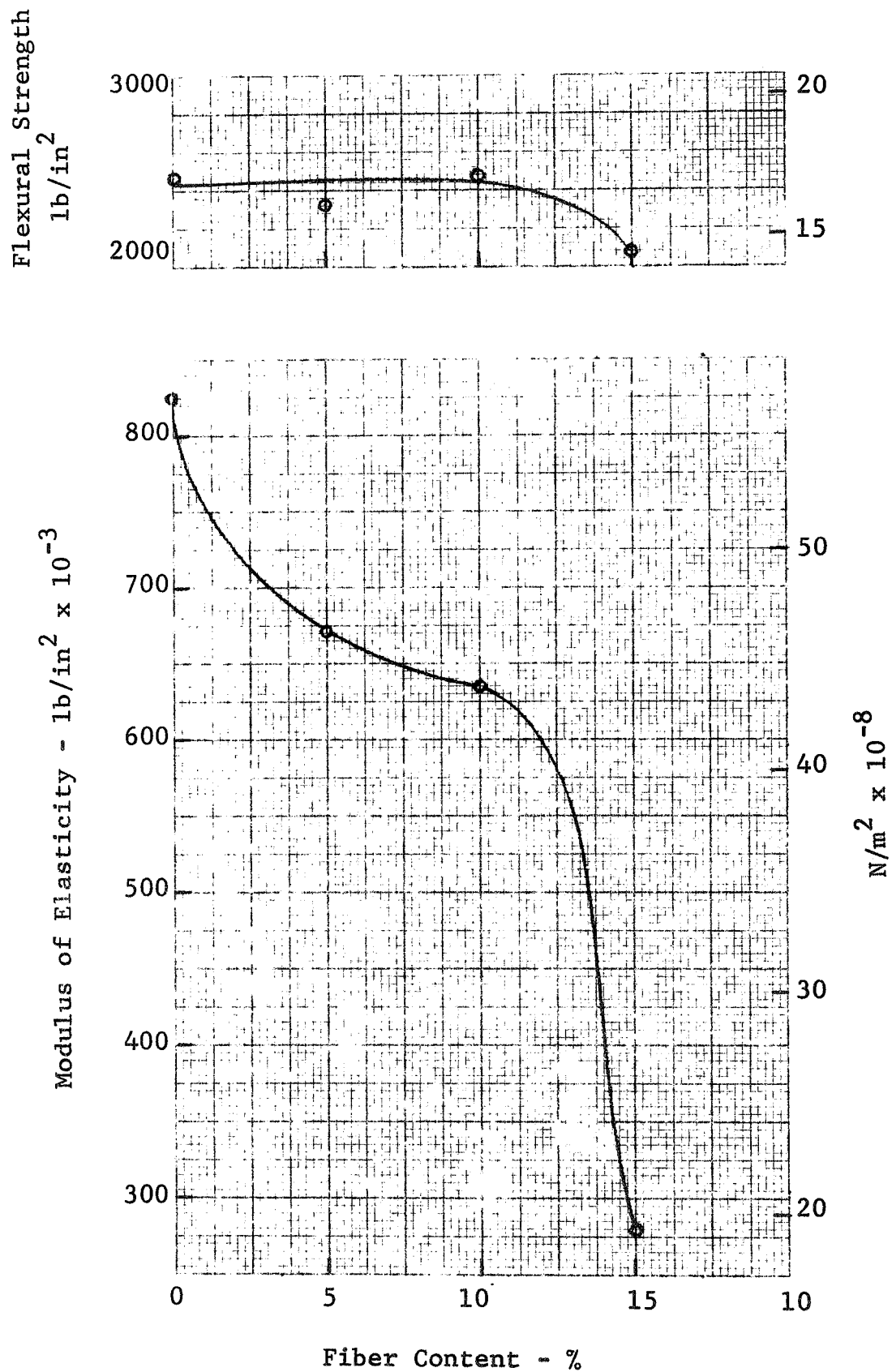


Figure 7 EFFECT OF FIBER CONTENT ON FLEXURAL STRENGTH AND MODULUS OF CFA SAMPLES COMPACTED AT 20 lb/in^2 (0.137 MN/m^2)

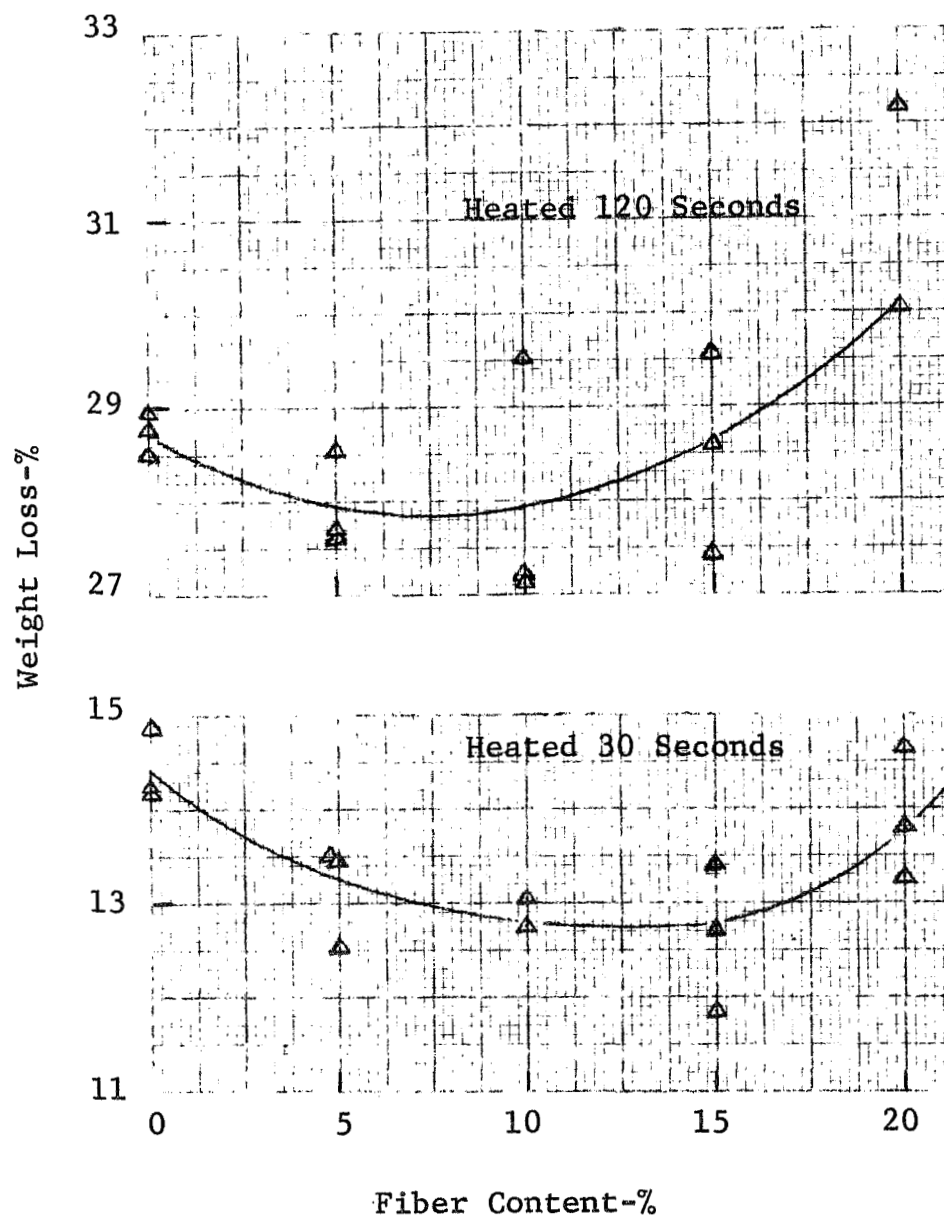


Fig. 8 WEIGHT LOSS VS FIBER CONTENT FOR HIGH-PRESSURE CFA SAMPLES HEATED 30 AND 120 SECONDS BY A PLASMA TORCH

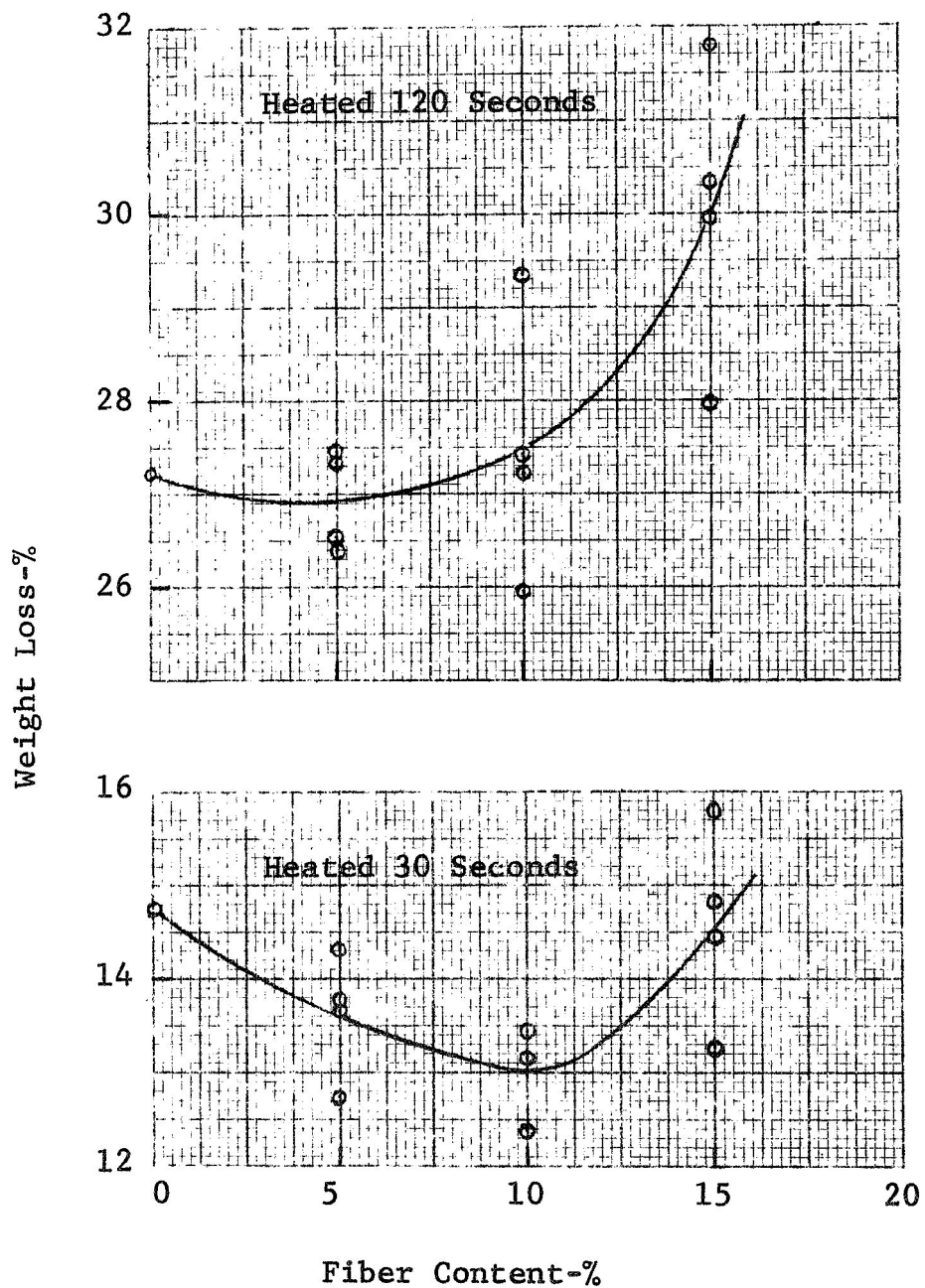


Fig. 9 WEIGHT LOSS VS FIBER CONTENT FOR LOW-PRESSURE CFA SAMPLES HEATED 30 AND 120 SECONDS BY A PLASMA TORCH

The flexural strength and modulus curves of reinforced samples all showed a minimum at a lower fiber content, which was interpreted to be the minimum-volume effect of classical composite theory. The initial fibers added to a matrix fail under low stresses because there are too few of them to restrain the elongation of the matrix. In this situation, the matrix must support the entire applied load with a cross sectional area reduced by the area of the useless fibers. The strength of the composite decreases until sufficient fibers are added that they are able to support part of the load. This concentration is known as the minimum volume. For the samples shown in Figure 5, the fiber content must approach 10% before the strength recovers to the level held when no fibers were used.

The deterioration of properties that sets in when the fiber content becomes so high that voids within undispersed clumps of fibers are present in appreciable quantities was not observed with the high-pressure samples, but it was clearly present in those compacted at low pressures (Figure 7). The strength was essentially constant from 0 to 10% fibers and dropped off beyond this range. The reason for this is obviously the low degree of compaction. The modulus of elasticity decreased sharply with increasing fiber content. The fiber-matrix bond was probably entirely mechanical, with the strength again dependent upon the compaction of the material. The porosity of these samples increased greatly with fiber content, which would also tend to lower the modulus.

c. Plasma Torch Erosion

(1) Weight Loss During Firing

Since the purpose of plasma torch tests was to rapidly screen the effects of changes in formulations and techniques, variations were determined simply by weighing the specimens. Measurement of the erosion of the curved surface produced by the torch was less accurate, so relatively little of this information was obtained.

The effect of carbon fibers on weight loss during a plasma torch test is shown in Figures 8 and 9. All four curves pass through a shallow minimum near 10% fibers. The weight loss changes in the range of fiber contents investigated were so small (2 or 3%) that these minima were not considered to be of major significance in selecting an optimum fiber concentration.

The primary reason for incorporating cut fibers in the CFA mix is to maintain the structural integrity of samples during the thermal shock and erosion of the plasma torch test. The specimens reported in the preceding paragraph were quite successful in this respect. Close examination of the heated surfaces revealed some fine, short cracks, but the overall appearance was one of a sound structure. This contrasts with the network of wide cracks observed in non-reinforced samples from previous tests. Comparative photographs of specimens with and without fibers are presented in Figure 10.

Figure 11 shows the weight loss as a function of binder content for a large number of samples. A linear curve was drawn for each heating period. Because there was a fair degree of scatter in the data, no independent correlations could be made for fabrication pressure, formulation or other variables.

Samples from a group cut from one molded plate were heated in the plasma torch for various periods from 15 to 135 seconds and weighed after cooling. The data are plotted in Figure 12. The rate of weight loss is seen to decrease between 0 and 60 seconds and become constant thereafter. In the curved section, the volatile constituents are being driven from the material, and in the straight section the carbon is being eroded at a constant rate of 9% (weight) per minute. Figure 13 shows the results of a similar test on a series of low-pressure samples. The variable section is longer, but the curve becomes straight at about 105 seconds.

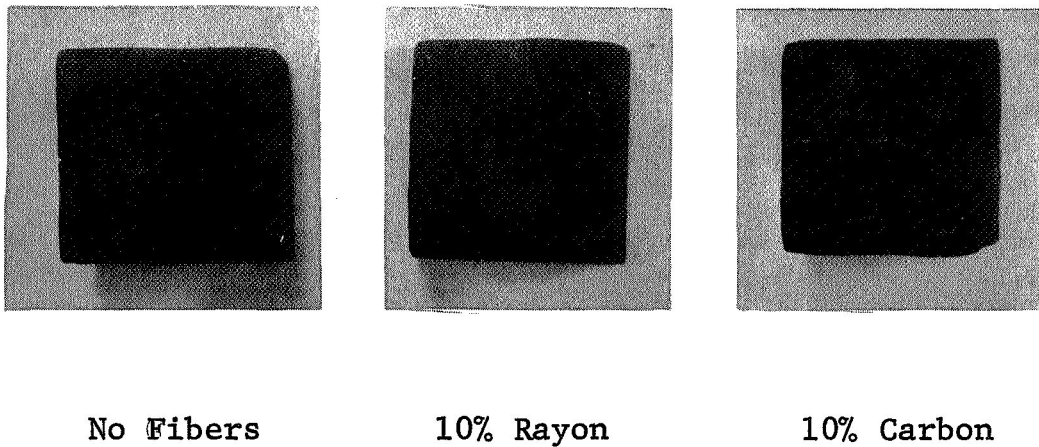


Fig. 10 EFFECT OF RAYON AND CARBON FIBERS IN
MAINTAINING STRUCTURAL INTEGRITY OF
CFA SAMPLES HEATED 120 SECONDS AT 5800°F
(3204°C) BY A PLASMA TORCH

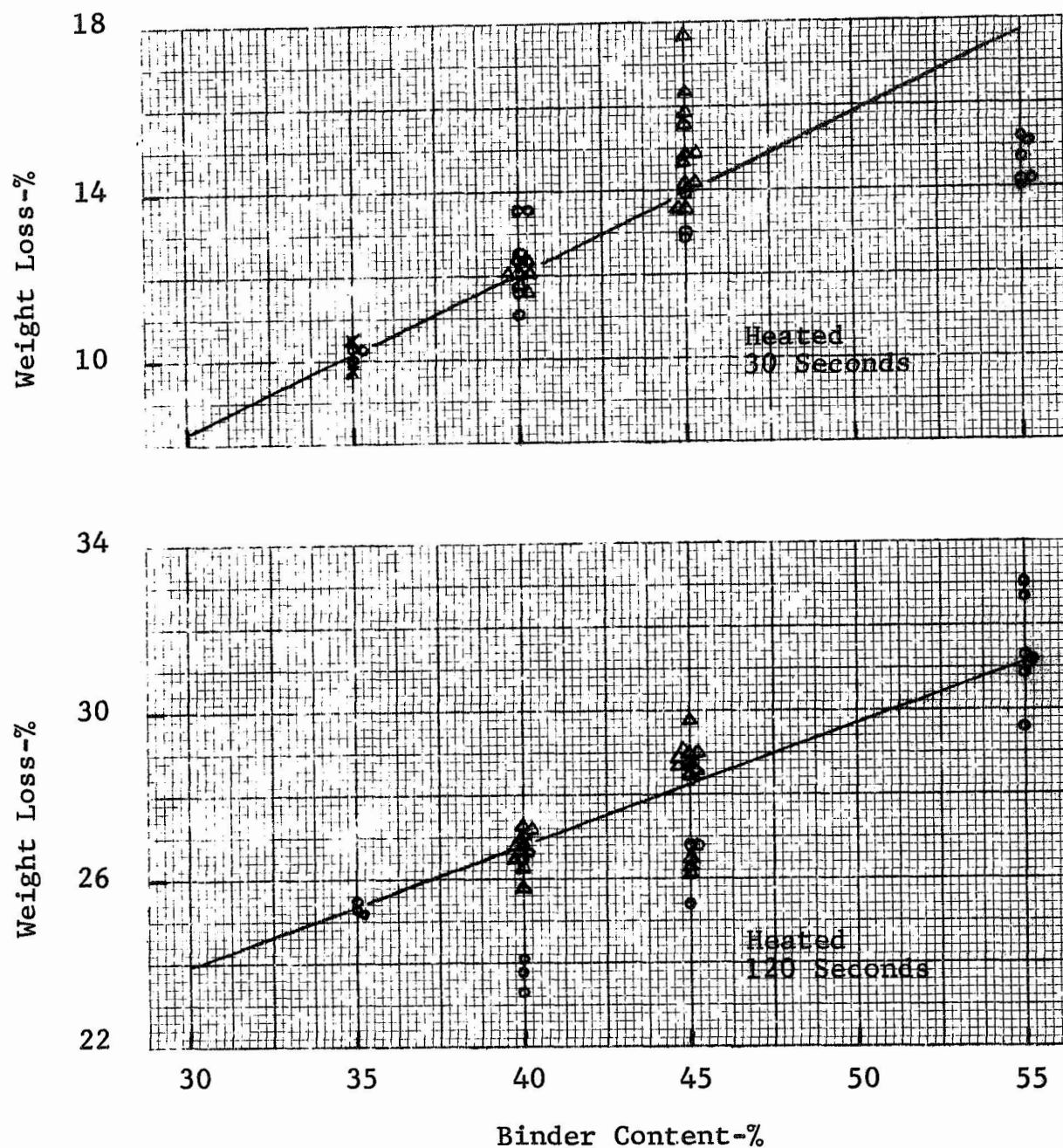


Fig.11 WEIGHT LOSS VS. BINDER CONTENT FOR CFA SAMPLES
HEATED 30 AND 120 SECONDS BY A PLASMA TORCH
(° = Low Pressure, Δ = High Pressure)

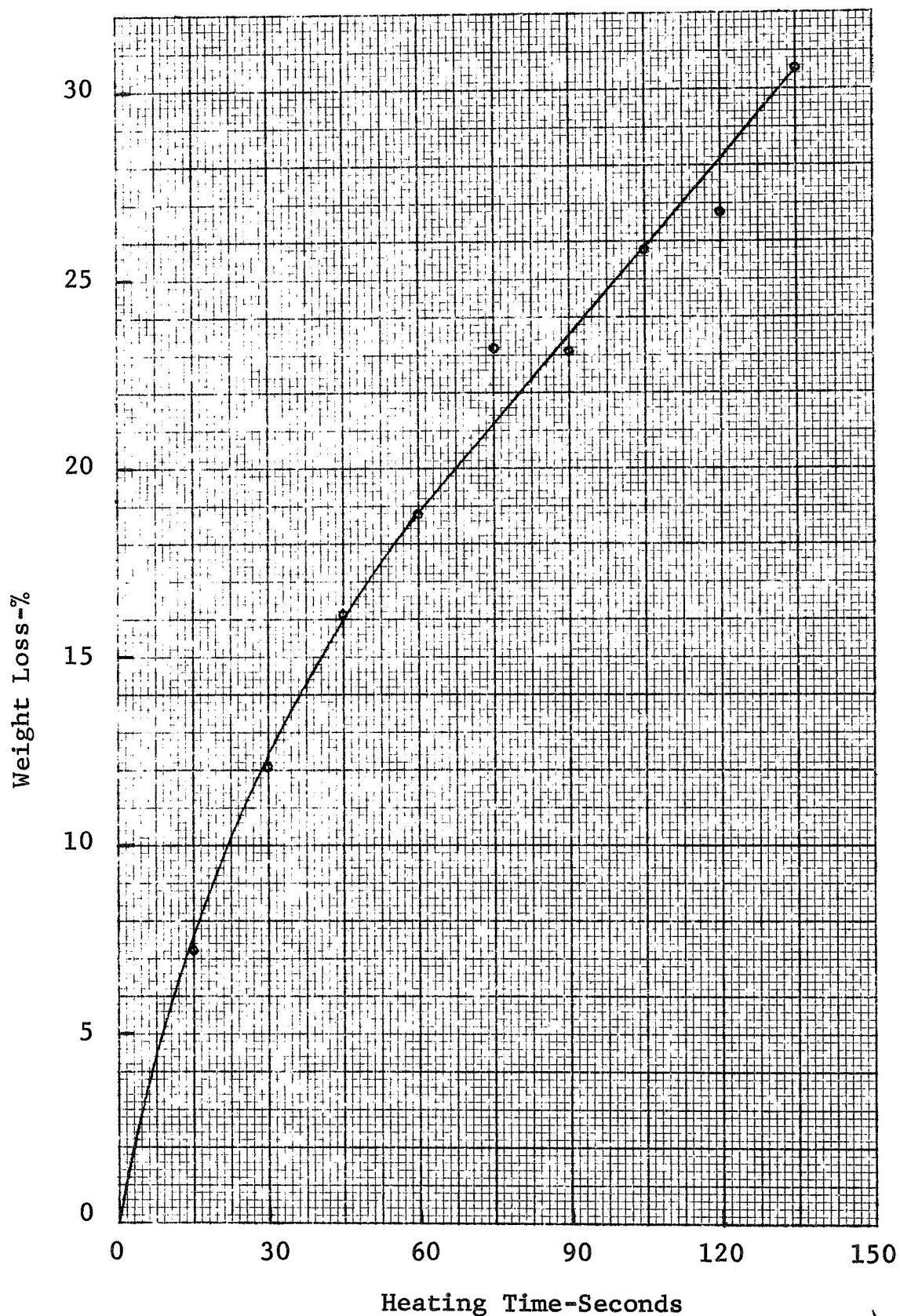


Fig. 12 RATE OF WEIGHT LOSS OF A HIGH-PRESSURE CFA SAMPLE HEATED BY A PLASMA TORCH (Sample 19473-13-6)

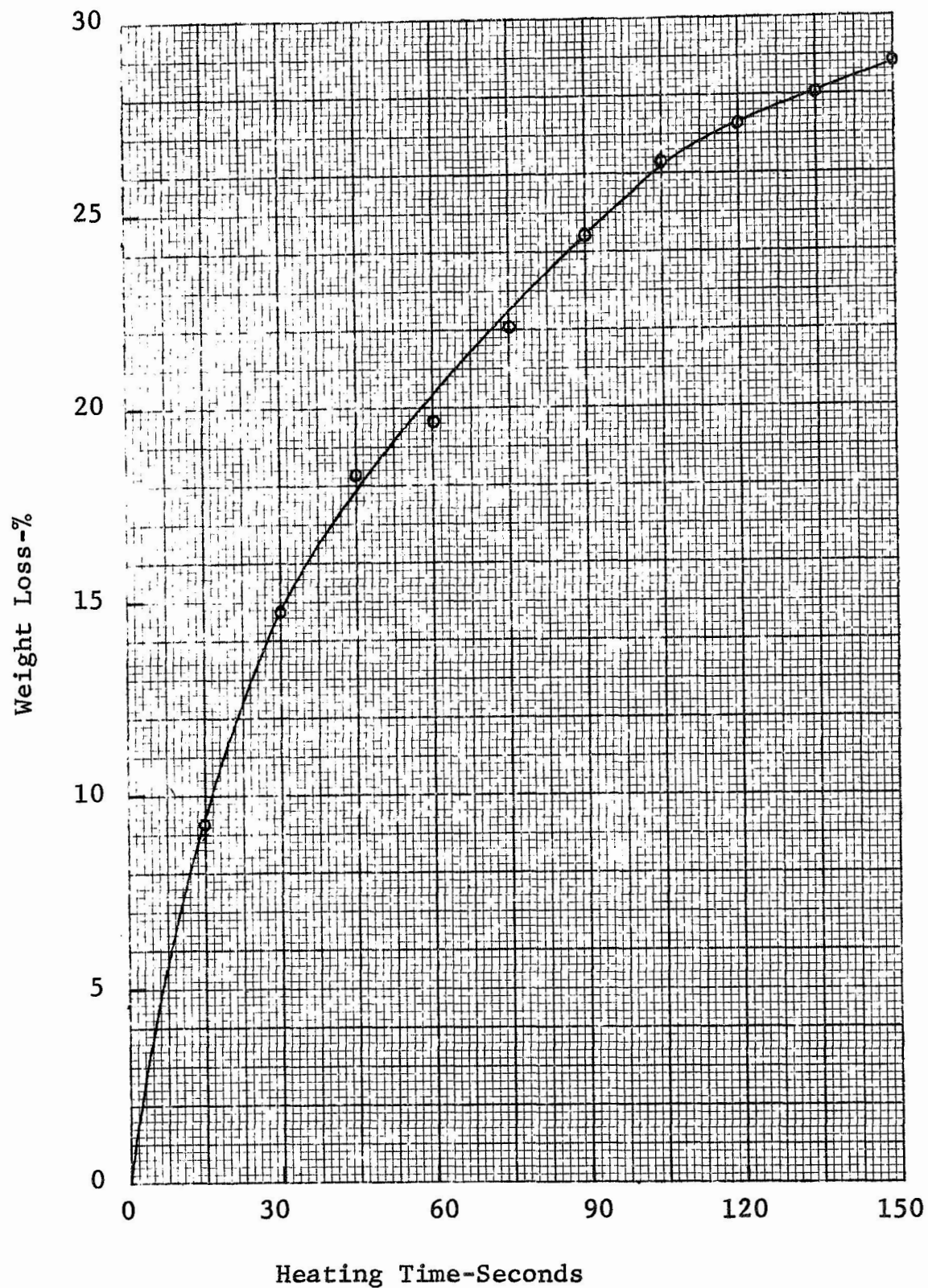


Fig. 13 RATE OF WEIGHT LOSS OF A LOW-PRESSURE CFA SAMPLE HEATED BY A PLASMA TORCH (Sample 19546-18-1)

(2) Temperature Gradient in a CFA Structure

A special test was conducted to determine the rate of heating in a specimen at various distances behind the heated face. Blocks 1-in. x 1-in. x 1-1/2-in. (2.54cm x 2.54cm x 3.81cm) deep cut from a low-pressure billet were drilled to accomodate short thermocouples. CFA material was packed around the thermocouples and cured to eliminate external temperature gradients. Each sample was heated by the plasma torch in the usual manner, while time and temperature were read. The curves in Figure 14 illustrate the change of temperature with time between 5/16 and 1-in. (0.8 and 2.54cm) away from the hot face. The graph shows, for example, that after 60 seconds of heating, the temperature 1-in. (2.54cm) inside the sample has reached only 158°F (70°C), while the corresponding surface temperature for this run was 5800°F (3204°C). This information indicates that a nozzle liner an inch thick (plus safety factor) would be sufficient to protect the steel shell during firing. No separate insulation layer would be required.

(3) Surface Erosion

The loss of material from the heated surfaces of a series of specimens, in terms of mils/sec (or $\mu\text{m}/\text{sec}$) change in thickness, was determined and plotted vs. time in Figure 15. The erosion rate was quite low, which may be characteristic of the particular test conditions. The rate did not exceed 1 mil/sec (25 $\mu\text{m}/\text{sec}$) until the samples had been heated for 75 seconds.

(4) Evaluation of Rayon Yarn as a Reinforcement

Since the carbon yarn used in this work was obtained by pyrolyzing rayon yarn, it seemed logical to investigate the use of rayon as a reinforcement for the CFA mixture. If these fibers were able to hold a body together during a plasma torch test nearly as well as the carbon fibers, they would be a valuable replacement. The cost advantage would be substantial — price ratio carbon/rayon is 130.

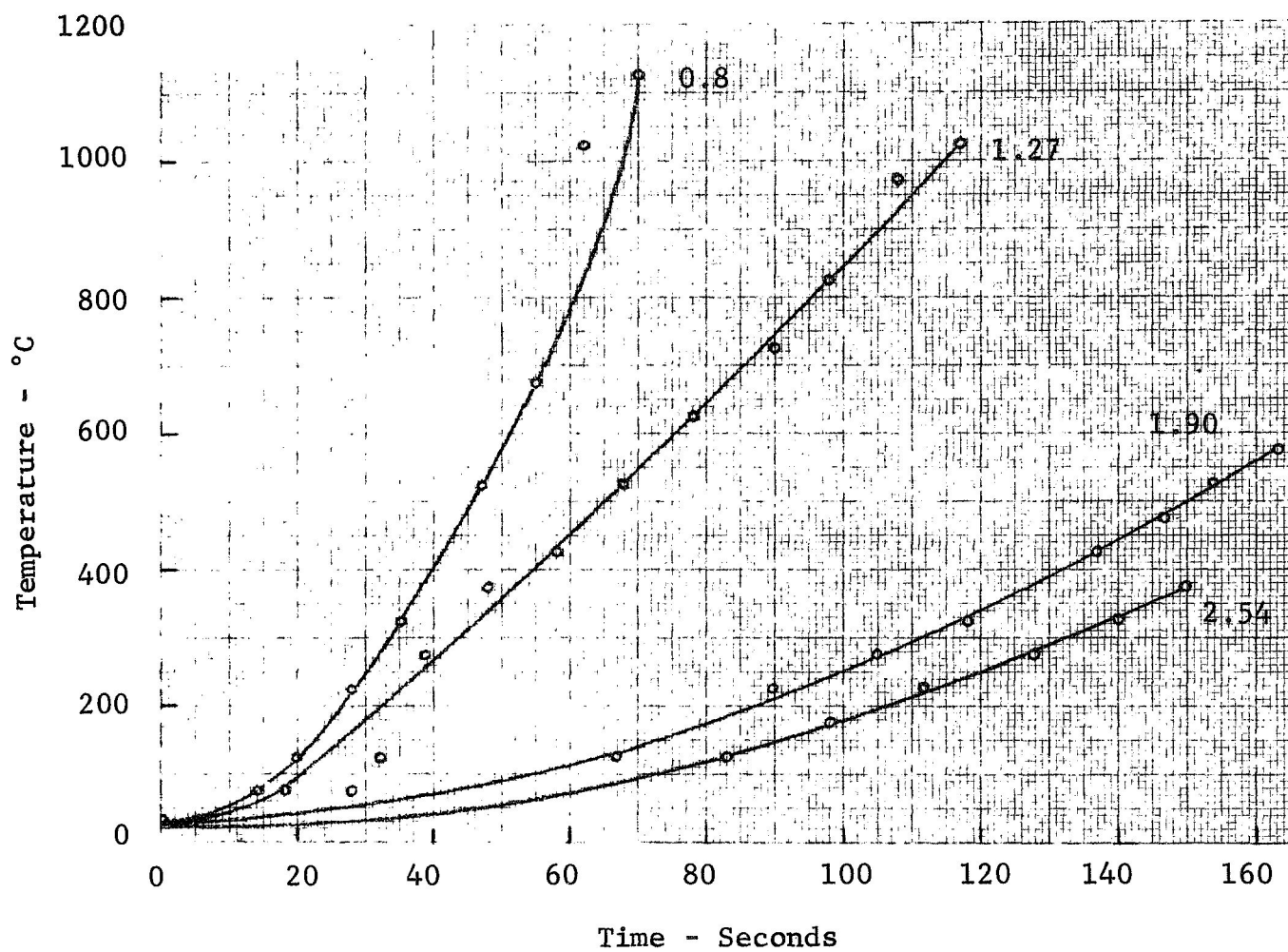


Fig. 14 HEATING RATE OF A SPECIMEN IN A PLASMA TORCH TEST
AT VARIOUS DISTANCES (cm) BEHIND THE FRONT FACE

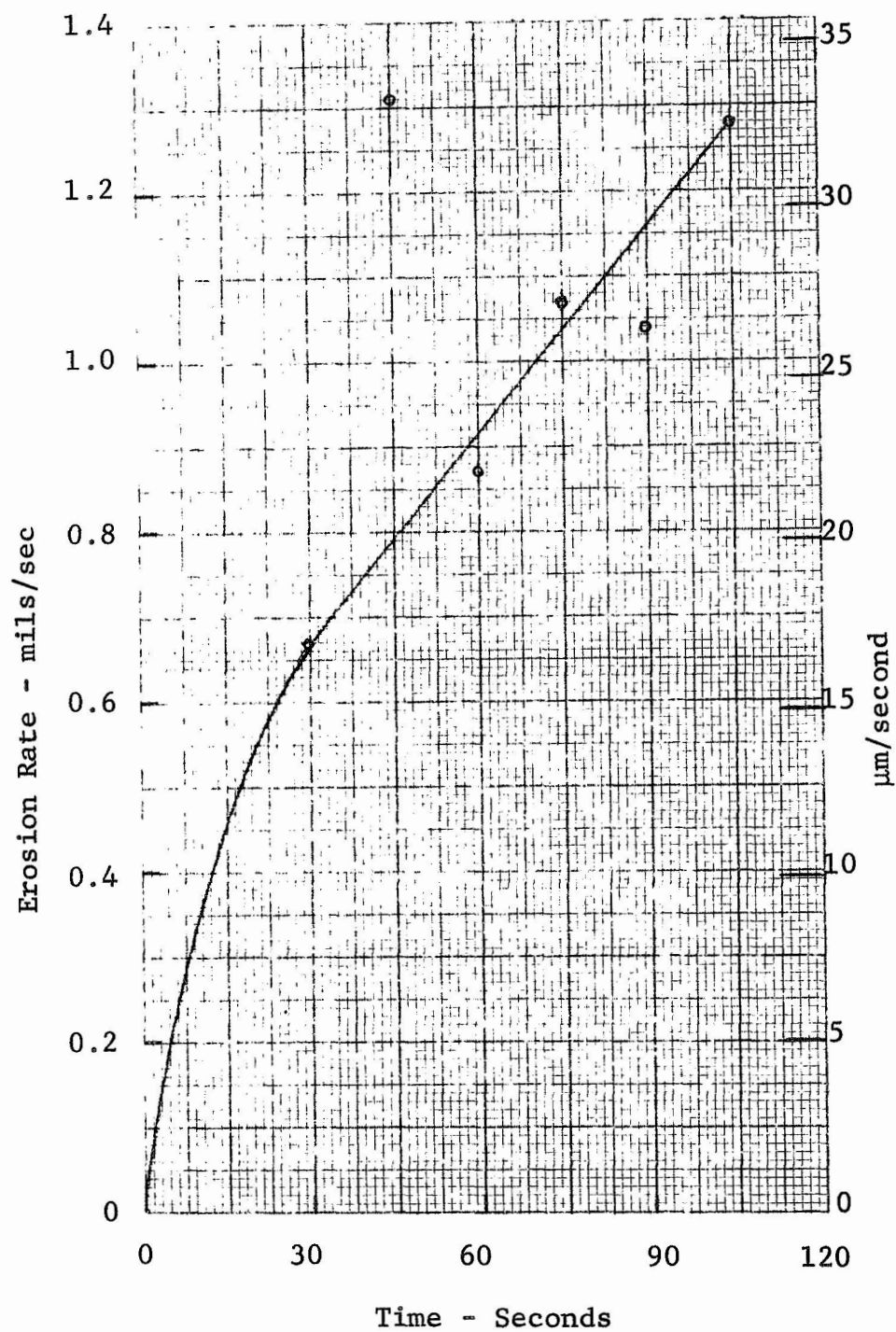


Fig. 15 EROSION RATE OF A LOW-PRESSURE CFA
SAMPLE HEATED BY A PLASMA TORCH
(Sample 19546-18-1)

A quantity of heavy rayon yarn (220 denier) was coated with binder and processed exactly like carbon yarn. A CFA mixture was prepared which contained 10% of these fibers and 33% binder. It was pressed at 32 lb/in² (0.22 MN/m²) and the resulting block showed normal bulk density and porosity for a body with 10% fibers (Table VII). In the plasma torch test, the specimens lost considerably more weight than would be expected for comparable pieces containing carbon yarn. The excess loss was attributed to volatiles escaping from the rayon yarn. The appearance of the samples was significant. Figure 10 shows the heated surfaces of specimens with no fibers, 10% rayon and 10% carbon. It is apparent that the rayon is an improvement over no reinforcement, but it is not quite as good as carbon. The limited time available precluded further investigation of rayon or any other low-cost organic yarn. This is definitely a promising technique which deserves further consideration.

C. Optimum Composition

The purpose of the Material Development Studies was to produce an optimum formulation for the CFA system which would meet the requirements for a rocket nozzle liner. Based upon all accumulated data, the formulation listed below was considered to offer the best combination of properties for the application.

Petroleum coke	52%
Carbon black	5
Carbon fibers	8
Binder	35
	<hr/>
	100%

The experimental results which lead to the selection of these ingredients and proportions are presented in the schematic plots of Figure 16. The best filler was found to be a very fine particle size petroleum coke, whose properties were enhanced by the addition of some carbon black. As shown in sections (a) to (d) of Figure 16, the shrinkage, density and flexural strength

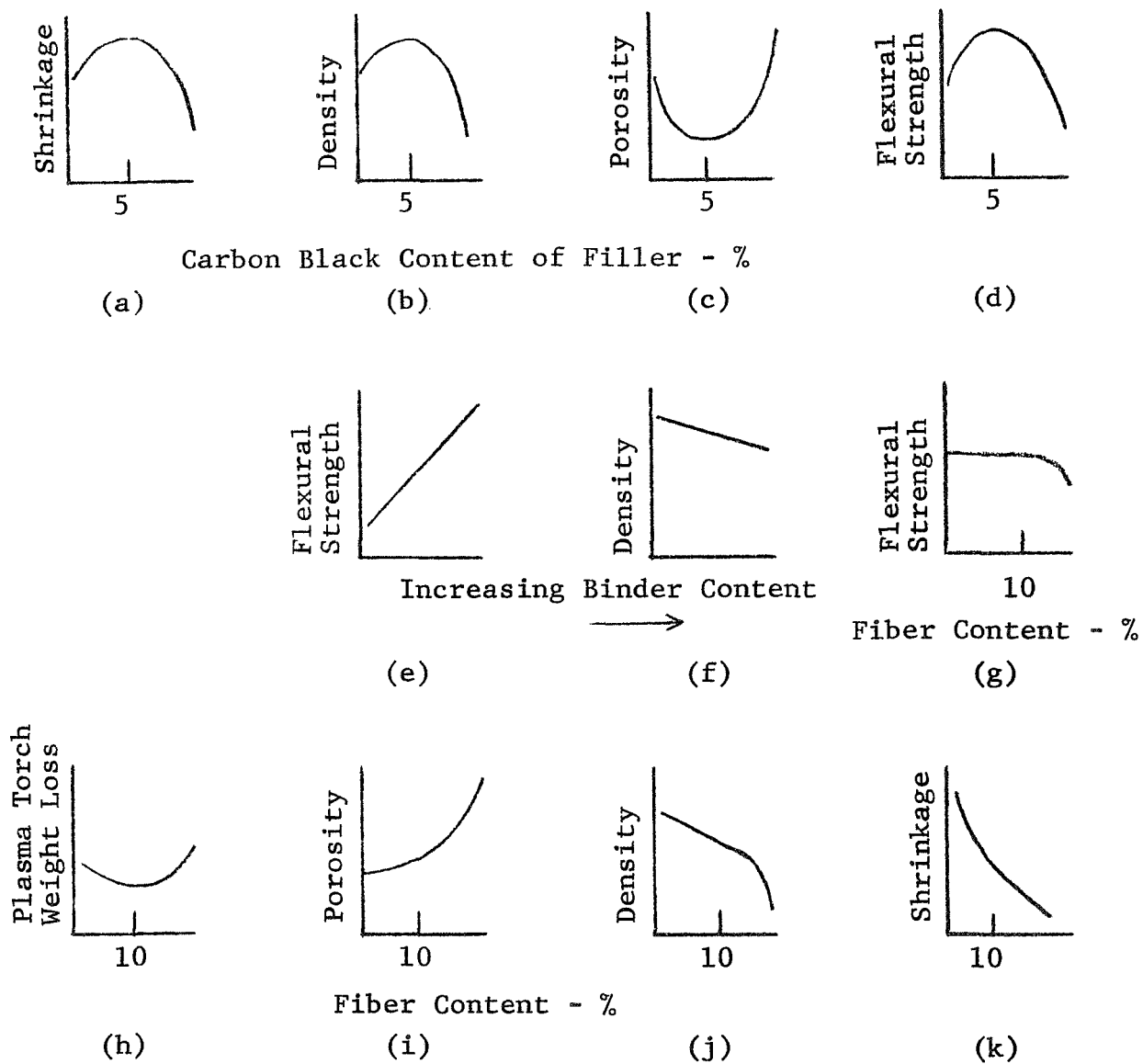


Fig. 16 SCHEMATIC GRAPHS ILLUSTRATING THE EFFECTS OF FILLER, BINDER AND FIBERS ON THE PROPERTIES OF CFA MIXTURES.

of CFA mixtures containing 40% binder and no additives reach a peak at a carbon black content of about 5%. The porosity is minimum at this concentration.

Sections (e) and (f) show the effect of binder content on two important properties. The flexural strength increases sharply, suggesting the use of higher binder contents, while the density decreases slightly as more binder is used. Another consideration is the effect of fabrication pressure on the properties of a body. Above 40% binder the pressure was found to have little effect on properties, while below 40% it had a very substantial effect. The optimum binder content was 40% for the first batch of petroleum coke, which was used through nearly all the development program. This figure was subsequently reduced to 35% because of the small proportion of fine particles in the second batch of coke.

Sections (g) through (k) of Figure 16 show the effect of fiber content on several properties. The shrinkage decreases as the fiber concentration increases, encouraging the use of as much reinforcement as possible. However, the flexural strength and density drop and the porosity rises sharply just above 10% fibers. The weight loss from plasma torch erosion passes through a minimum at 10% fibers. The minimum volume effect, discussed previously, indicates a content of 10%. Although all the indicators pointed to a fiber content of 10%, this quantity turned out to be somewhat excessive from the standpoint of fiber bulk vs. CFA bulk during scale up experiments. Large billets with 10% fibers were more difficult to mix intimately and press to high density than others with slightly less fibers. Compaction experiments with large billets indicated that 8% fibers would be better than the 10% fiber content.

E. Economic Considerations

One of the requirements for the rocket nozzle inserts prepared under this program was low cost. The CFA materials system is considered to be inherently economical because the basic

ingredients are low cost, and additions or substitutions may be readily made. Table IX presents the estimated costs of fabricating 100 pounds (45.4kg) of CFA as used for the inserts submitted on this program, along with comparative costs for two recommended modifications. The laboratory figure is based upon actual prices for the quantities of ingredients purchased for this program, while the two production lists used prices for the moderately large quantities which would be required to prepare a pilot-plant or small-production quantity of rocket nozzles. Proportions were those given in the optimum composition in the preceding section.

It is apparent that the fibers are the key to the material costs. The CFA mix alone costs 75¢/lb (\$1.65/kg) in small quantity and 44¢/lb (97¢/kg) in large quantity. The use of carbon yarn adds \$5.44/lb (\$11.98/kg) to the materials, compared with 4¢/lb (8.8¢/kg) for rayon yarn. A fiber of intermediate cost is staple carbon (1/4-in, 0.64cm long) from the manufacturer of the continuous yarn. This would add \$1.56/lb (3.44/kg) to the cost of plain CFA. The most expensive step in fabrication is precoating of yarn with binder and cutting it into staple. A brief study at the end of the program indicated that sound bodies could be fabricated from mixtures containing fibers which had not been precoated. The bulkiness of the fibers requires a special technique for combining the ingredients so that the fibers become wetted by the binder. A few optimization experiments should establish the necessary conditions for using uncoated fibers. This has been assumed in the second and third columns of Table IX. Machining is a substantial percentage of the per-pound cost, which may be eliminated in some applications. Where dimensional tolerances are not extremely critical, the bodies may be removed from the shaped die and used without further processing.

The total costs given in Table IX show clearly that nozzle inserts or other objects can be prepared economically from the CFA system described in this report. The target figure of \$2.00/lb (\$4.41/kg) could be reached if the fabrication or machining costs could be reduced slightly.

Table IX
ESTIMATED COSTS FOR PRODUCING ROCKET NOZZLE INSERTS
BY THE LABORATORY METHOD ACTUALLY USED AND
TWO PROPOSED PRODUCTION METHODS

	Cost Per 100 Pounds (45.36kg) of Cured CFA ⁽¹⁾		
	Laboratory \$	Production 1 ⁽²⁾ \$	Production 2 ⁽³⁾ \$
CFA Material			
Petroleum coke	32.14	3.86	3.86
Carbon black	0.78	0.37	0.37
Furfuryl ester resin	37.85	37.85	37.85
2-Furaldehyde	2.57	0.58	0.58
Coal tar pitch	0.36	0.36	0.36
Catalyst (ZnCl ₂)	1.11	0.64	0.64
Fibers	543.84	155.74	4.16
Subtotal	618.65	199.40	47.82
Fabrication			
Fiber coating	500.00	—	—
Mixing and screening	100.00	50.00	50.00
Pressing	20.00	20.00	20.00
Curing	10.00	10.00	10.00
Subtotal	630.00	80.00	80.00
Machining and Inspection	1335.00	111.00	111.00
Total	2583.65	390.40	238.82
Cost per pound	\$25.84	\$3.90	\$2.39

Notes for Table IX

1. Weight of ingredients 103 lb. (46.7kg). Assumed weight loss during curing 3 lb. (1.36kg).
2. Production 1 is based upon the following assumptions:
 - a. Large quantities of chemicals will be used, with appropriately lower prices.
 - b. Commercial 1/4-in. (0.64cm) staple carbon fibers will be used in place of yarn.
 - c. The fibers will not be coated by a separate treatment.
 - d. Processing of the mix will be carried out in suitably large commercial equipment.
 - e. Insert blanks will be pressed to shape and reasonably close to final size.
 - f. Machining of blanks will be on a continuous basis.
3. Production 2 is based upon the same assumptions, except that the fibers will be commercial organic yarn, cut into staple but not separately coated. The price listed is for rayon yarn.

III. MATERIAL CHARACTERIZATION

The second phase of the program was the characterization of the optimum formulation by measurement of several physical and mechanical properties. A billet about 9-3/4-in. (24.8 cm) diameter by 5-in. (12.7 cm) thick was pressed so that all the required specimens could be cut from the same piece. The bulk density was 1.30 g/cc, which was the target value for the first set of nozzle inserts. The complete set of test specimens is illustrated in Figure 17. A brief description of test procedures and results is given in the following sections. The property values are presented in Table X.

A. Impact Strength

The impact strength was measured on an 18-in.-lb (20.7 cm-kg) swinging-pendulum machine with a charpy head. The test bars were 1/4-in. x 1/4-in. x 3-in. (0.64 cm x 0.64 cm x 7.62 cm), half with 45° notches and half plain. The notched specimens did not break at the notches, and all the fractured surfaces showed the fibrous appearance of a fractured wood beam. These observations and consideration of the impact strength values showed that this material was quite insensitive to the presence of notches. For comparison, samples of the same configuration were prepared from a commercial product (ATJ graphite) and tested on the same machine. The corresponding average readings were 0.86-in.-lb. (0.99 cm-kg) without notch and 0.23-in.-lb (0.26 cm-kg) with notch. Not only was the commercial material weaker, but it showed a strong sensitivity to the presence of notches.

B. Compressive Strength

The compressive strength was measured on a Riehle testing machine with a 5000-lb (2268 kg) load scale. The specimens were cylinders 1-in. (2.54 cm) diameter by 3-in. (7.62 cm) long. They failed in shear diagonally along most of the sample length. The average strength of 5066 lb/in² (34.9 MN/m²) compares with a value of 8500 lb/in² (58.6 MN/m²) for commercial ATJ graphite.

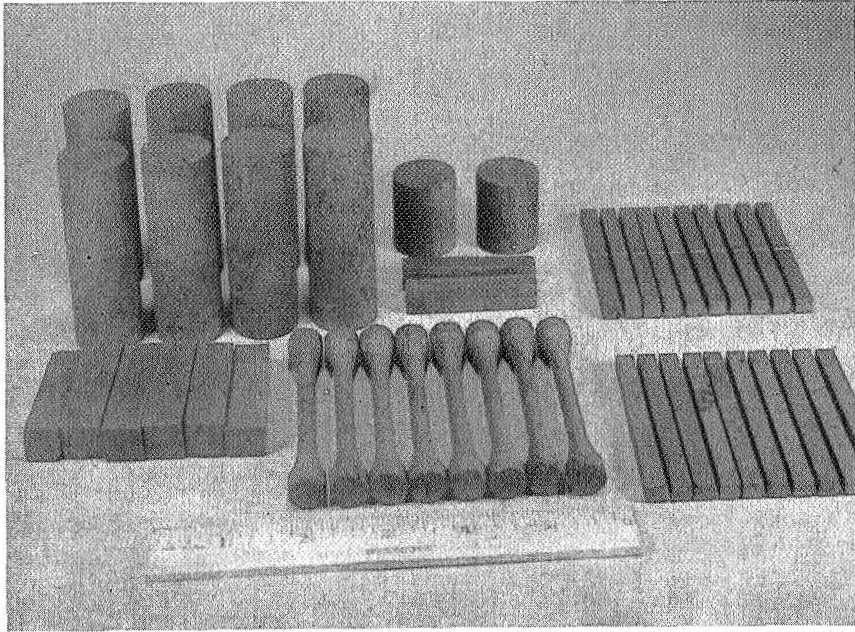


Fig. 17 MATERIAL CHARACTERIZATION SPECIMENS

Table X
RESULTS OF MATERIAL CHARACTERIZATION TESTS

Property	Samples	Average Value	Unit	Standard Deviation	Average Value	Unit	Standard Deviation
Impact Strength							
Without Notch	10	0.83	in-lb	0.43	0.96	cm-kg	0.50
With Notch	10	1.15	in-lb	0.48	1.33	cm-kg	0.55
Compressive Strength (WG)	6	5066	lb/in ²	364	34.9	MN/m ²	2.50
Tensile Strength (WG)	6	206	lb/in ²	49	1.42	MN/m ²	0.34
Shear Strength							
With Grain	5	702	lb/in ²	352	4.84	MN/m ²	2.42
Across Grain	4	184	lb/in ²	30	1.27	MN/m ²	0.21
Bulk Density	6	0.047	lb/in ³	0.001	1.30	g/cc	0.03
Apparent Density	6	0.062	lb/in ³	0.001	1.71	g/cc	0.02
Porosity	6	24.1	%	2.7	-	-	-
Thermal Expansion	2	27.4	in/in/ °F x 10 ⁶	-	49.2	cm/cm/ °C x 10 ⁶	-
Thermal Conductivity	1	0.65	BTU/hr- ft ² -°F/ft	-	0.0027	Cal/sec- cm ² -°C/cm	-

This is a favorable comparison in consideration of the respective densities: 1.30 vs. 1.73 g/cc.

C. Tensile Strength

Tensile strength was measured on the Riehle testing machine with a 250-lb (113-kg) load scale. The specimens were cylindrical ("dogbones") with a diameter of 0.5 in. (1.27 cm) in the grips and 0.25 in. (0.64 cm) in the gauge length. Spherical collet grips were used to align the axis of applied stress with the axis of the specimen. The average strength was rather low, because of the low density of the optimized composite material. It is this material which exhibited the best overall combination of properties during screening tests.

D. Shear Strength

Shear strength was measured on the Riehle testing machine with a 500-lb (226 kg) load scale. The 1/2-in. x 1/2-in. x 2-in. (1.27-cm x 1.27-cm x 5.08-cm) strips were broken in a short-beam flexure setup with three-point loading and a support span of 1-in. (2.54 cm). Because of the short length, some strips were cut from the billet horizontally (WG, with grain) and some were cut vertically (AG, across grain). Those designated WG had the benefit of reinforcement by the carbon fibers, while the AG types were fractured along the fibers and thus received no reinforcement. The anisotropy ratio of strengths, WG/AG, was 3.8, showing the effect of the fibers in strengthening the material in one direction.

E. Density-Porosity

The density and porosity were determined by the method described in Section IIB. The sample size was 1/2-in. x 1/2-in. x 3-1/2-in. (1.27 cm x 1.27 cm x 8.90 cm). The results were considered normal compared with those obtained from material-development specimens.

F. Thermal Expansion

Thermal expansion was measured with an electronic dilatometer which produced a graph of temperature vs expansion on an XY Plotter. The samples were 1/4-in. x 1/4-in. x 1-in. (0.64 cm x 0.64 cm x 2.54 cm) strips, and the heating rate of the furnace was 9°F/min (5°C/min). When the samples were heated, the shrinkage accompanying the removal of volatiles from the binder exceeded the expansion of the material from the temperature rise. The curve reached a maximum at about 437°F (225°C) and dropped below the initial setting (zero expansion) at about 617°F (325°C). A second run on one of the samples showed a similar maximum, but at a much higher temperature 842°F (450°C). The coefficient of expansion for the second run was much lower - 11.4×10^{-6} cm/cm/°C.

G. Thermal Conductivity

Thermal conductivity was measured on one sample at 302°F (150°C) by the split-block technique. The result was very low for carbon because of the high porosity of the body and the low conductivity of the furfuryl ester resin.

IV. NOZZLE FABRICATION

A rocket nozzle design was provided by NASA-Lewis for evaluation of the CFA material under actual use conditions. It consisted of a three-piece structure - entrance, throat and exit cones - with a throat diameter of 1.8-in. (4.57 cm) and an expansion ratio of 7 (Figure 18). Two assemblies were required for testing, the first to be fired and evaluated before the second was fabricated. Two cylindrical dies were prepared for compacting the CFA mix at low pressures (10 to 60 lb/in², 0.069 to 0.41 MN/m²).

For the first assembly, several billets were pressed in the manner used for the small test pieces in the material-development program. Because of differences in the construction of the large and small dies, the billets were able to expand more when the load was removed. Within a few hours of pressing, these samples increased 1/2-in. to 3/4-in. (1.27 cm to 1.91 cm) in length (in the pressing direction). Customarily they were placed in an oven the day after pressing and cured without an applied load. The actions of heating and movement of volatiles through the body offered another opportunity for the material to expand. Consequently, it was difficult to attain the desired density of 1.30 g/cc. One billet was repeatedly re-pressed over a 3-day period (before curing), which resulted in some improvement. The final cured density reached 1.37 g/cc.

Three billets were selected and machined into the three nozzle inserts. Because of the difficulty described above, many of the outside edges tended to be rounded rather than sharp. The inner edges, which are more critical, were satisfactorily sharp. Some data on the first set of inserts are presented in Table XI.

The firing test of the first nozzle assembly showed an erosion rate slightly higher than would be desirable (7.95 mils/sec or 202 μ m/sec). Therefore the second set of billets was fabricated to a higher density. To accomplish this, a modification of the technique was adopted. The size of the pressing setup precluded compacting the CFA material in an oven, so a battery of heat lamps

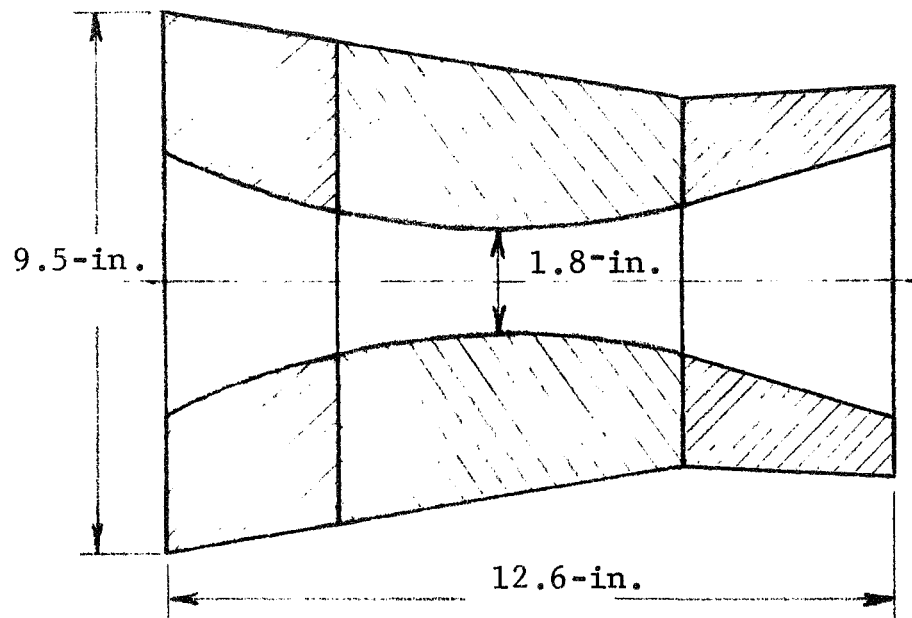
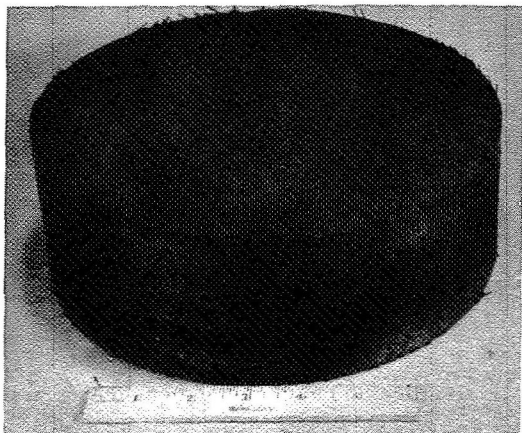


Fig. 18 SCHEMATIC CROSS SECTION OF
NOZZLE ASSEMBLY

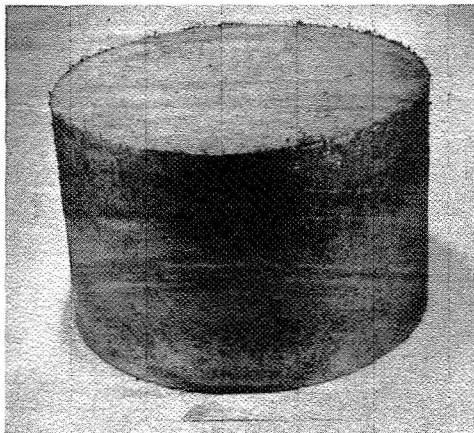
Table XI
PROPERTIES OF NOZZLE INSERTS

Assembly	Insert	Insert Number	Fabrication Pressure ²		Weight g	Bulk Density g/cc
			lb/in ²	MN/m ²		
1	Entrance	19828-12-1	28	0.193	3166	1.14
	Throat	19828-12-2	33	0.228	5718	1.37
	Exit	19828-12-3	36	0.248	1808	1.27
2	Entrance	19948-8-1	13	0.090	3990	1.50
	Throat	19948-8-2	20	0.138	6180	1.55
	Exit	19948-8-3	36	0.248	2203	1.58

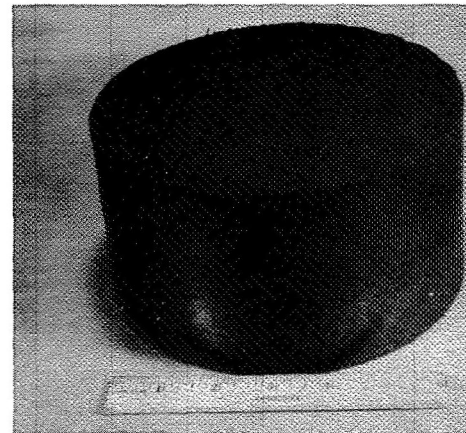
was mounted around the die. The pressing operation was extended from a few minutes to 4 hours - 2 hours of heating and 2 hours of holding at a temperature slightly above 212°F (100°C). This was sufficient to cure the resin hard and increase the density to 1.50 g/cc or more. After removal from the die, the billets were heated slowly to 302°F (150°C) and finish cured in the usual manner. The machined inserts showed smooth surfaces and all sharp edges. The second set of billets and inserts is illustrated in Figure 19. The assembled nozzle is shown in Figure 20. Data on the inserts are given in Table XI.



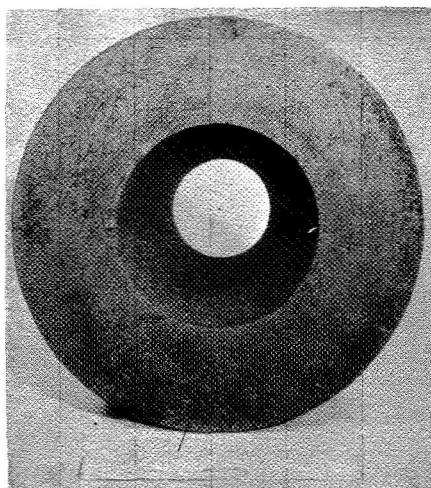
Billet 19828-24-1



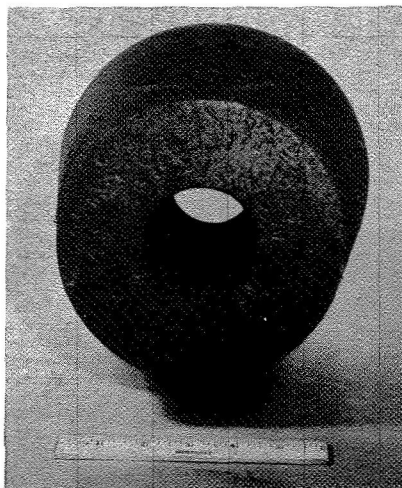
Billet 19948-5-1



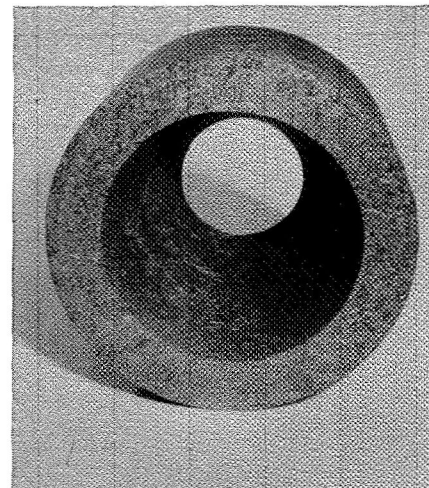
Billet 19828-19-1



Entrance Insert 19948-8-1



Throat Insert 19948-8-2



Exit Insert 19948-8-3

Fig. 19 BILLETS AND CORRESPONDING INSERTS FOR THE SECOND NOZZLE ASSEMBLY

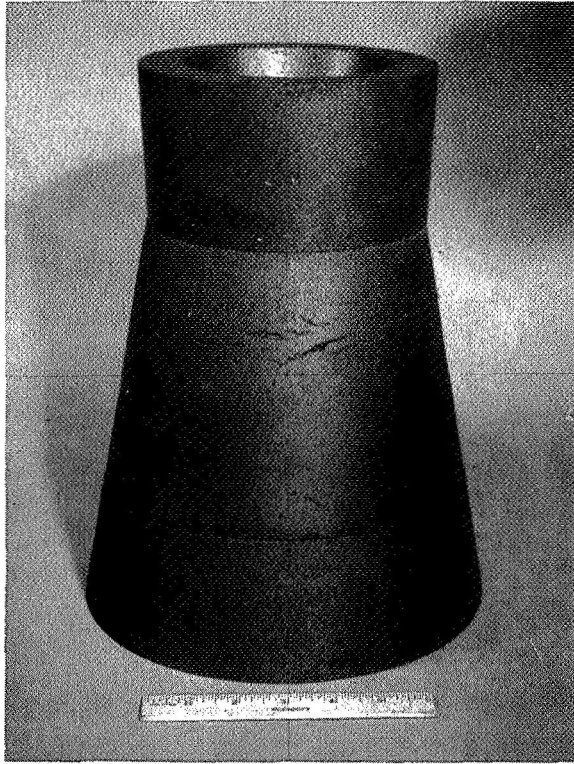


Fig. 20 SECOND NOZZLE ASSEMBLY BEFORE FIRING

V. POST-TEST ANALYSIS

The nozzles were test fired by the Aerojet Solid Propulsion Company, Sacramento, California. After completion of the tests, the internal diameters were measured at three positions around the circumference and 12 stations along the axis of each nozzle to determine the depth of material lost by erosion. The data are shown in Tables XII and XIII. The average depth was divided by the firing time to determine the average rate of erosion. The rate was then adjusted to a constant chamber pressure of 600 lb/in^2 (4.14 MN/m^2) for comparative purposes.

Profiles of the depth of erosion are shown on cross sections of the nozzles in Figure 21. The difference in material loss is evident, the higher density second nozzle showing lower and more uniform erosion. Material loss in the exit cones was small and approximately the same in each case, even though the density values differed considerably. This shows that a lower density body may be safely used in the exit, if desired. The lower limit on density would be about 1.20 g/cc in order that the insert have practical machining and handling characteristics. The entrance and throat sections must be pressed to relatively high densities (1.50 to 1.60 g/cc) to resist the severe temperature and pressure conditions during rocket firing.

The records of motor chamber pressure vs time are shown in Figures 22 and 23. The dips in the curves were due to variations in the rate of burning at adhesive joints between sections of the propellant charge. The firing times were 48.2 seconds for nozzle 1 and 39.2 seconds for nozzle 2, and the average chamber pressures were, respectively, 384 and 515 lb/in^2 abs. (2.64 and 3.55 MN/m^2). Figure 24 shows the appearance of all the inserts after testing. The improved erosion resistance of the higher density nozzle 2 is clearly evident. Cross sections of the fired nozzles are illustrated in Figure 25.

Table XIV presents comparative data for rocket nozzles made from various forms of carbon and tested by the same contractor

Table XII
EROSION OF NOZZLE ASSEMBLY 1
DURING FIRING IN A ROCKET MOTOR

	ENTRANCE			THROAT						EXIT		
Station ⁽¹⁾	12	11	10	9	8	7	6	5	4	3	2	1
Area Ratio ⁽²⁾	4.04	3.23	2.63	1.48	1.15	1.01	1.05	1.28	1.76	2.60	4.00	5.35
Erosion } 0° ⁽³⁾	305	225	455	180	305	200	215	115	20	75	30	15
Depth, } 120°	350	295	380	250	250	270	175	135	80	80	25	25
mils } 240°	415	480	650	365	340	335	300	210	95	90	25	20
Average Depth, mils	357	333	495	265	298	268	230	153	65	81	27	20
Rate, mils/sec ⁽⁴⁾	7.41	6.91	10.27	5.50	6.18	5.56	4.77	3.17	1.35	1.68	0.56	0.42
Adjusted Rate ⁽⁵⁾	10.58	9.88	14.67	7.86	8.84	7.95	6.83	4.54	1.93	2.41	0.81	0.60

- (1) Locations of stations are shown in Figure 21.
(2) Ratio of cross sectional area of nozzle at a given location to the minimum area in the throat.
(3) Erosion measured at three angles around the inner circumference of the nozzle.
(4) Firing time 48.2 seconds.
(5) Erosion rate adjusted to 600 psia chamber pressure by the formula

$$\text{Adjusted Rate} = (\text{Actual Rate})(600/\text{Actual Pressure})^{0.8}$$

Table XIII
EROSION OF NOZZLE ASSEMBLY 2
DURING FIRING IN A ROCKET MOTOR

	ENTRANCE			THROAT						EXIT		
Station (1)	12	11	10	9	8	7	6	5	4	3	2	1
Area Ratio (2)	4.04	3.23	2.63	1.48	1.15	1.01	1.05	1.28	1.76	2.60	4.00	5.35
Erosion } 0° (3)	72	85	150	126	140	210	150	80	15	45	30	20
Depth, } 120°	80	95	142	175	175	189	175	93	60	55	35	5
mils } 240°	92	170	197	255	234	238	210	147	65	65	35	20
Average Depth, mils	81	117	163	185	183	212	178	107	47	55	33	15
Rate, mils/sec (4)	2.07	2.99	4.17	4.73	4.68	5.42	4.55	2.74	1.20	1.41	0.85	0.38
Adjusted Rate (5)	2.34	3.47	4.72	5.35	5.29	6.13	5.15	3.10	1.36	1.59	0.96	0.43

(1) Locations of stations are shown in Figure 21.

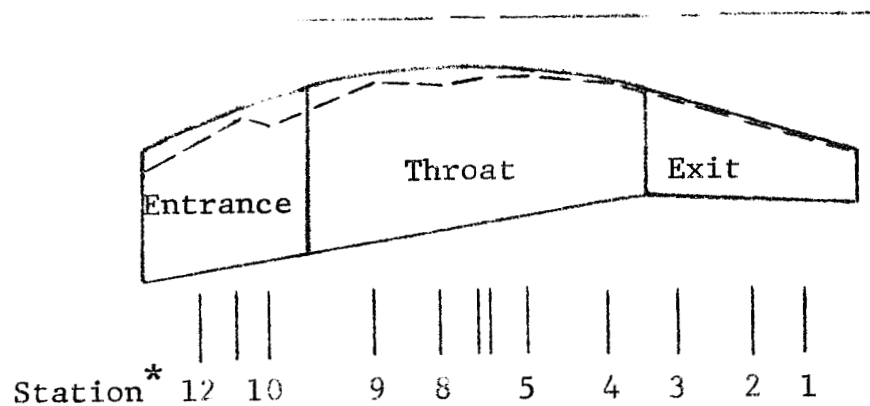
(2) Ratio of cross sectional area of nozzle at a given location to the minimum area in the throat.

(3) Erosion measured at three angles around the inner circumference of the nozzle.

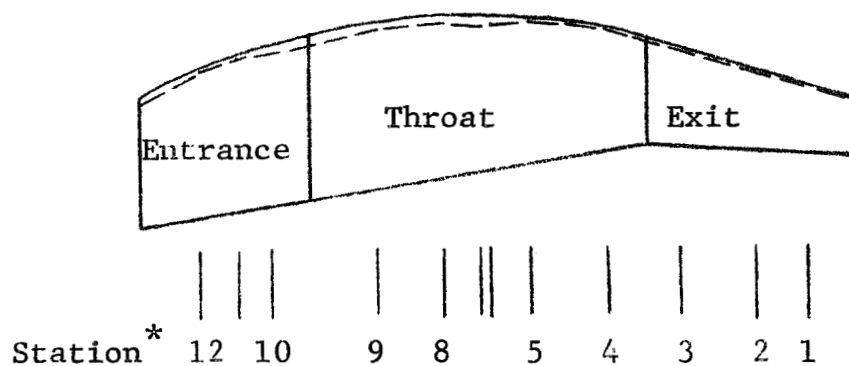
(4) Firing time 39.2 seconds.

(5) Erosion rate adjusted to 600 psia chamber pressure by the formula

$$\text{Adjusted Rate} = (\text{Actual Rate})(600/\text{Actual Pressure})^{0.8}$$



NOZZLE 1



NOZZLE 2

Fig. 21 EROSION PROFILES FOR FIRED NOZZLES (At 0° Position)

*Nozzle stations are the same as referenced in firing tables.

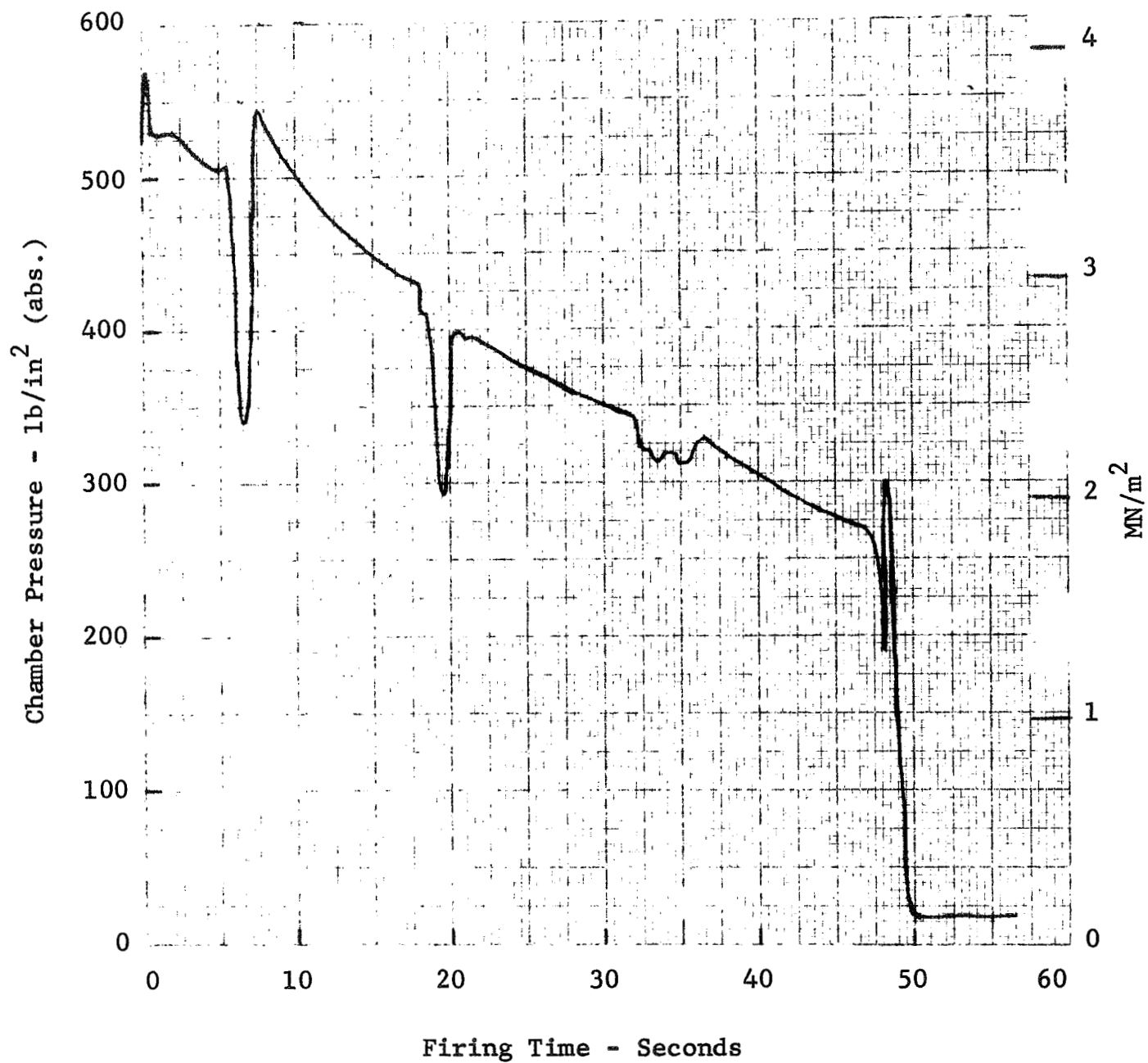


Fig. 22 VARIATION OF CHAMBER PRESSURE WITH TIME DURING FIRING OF NOZZLE 1.

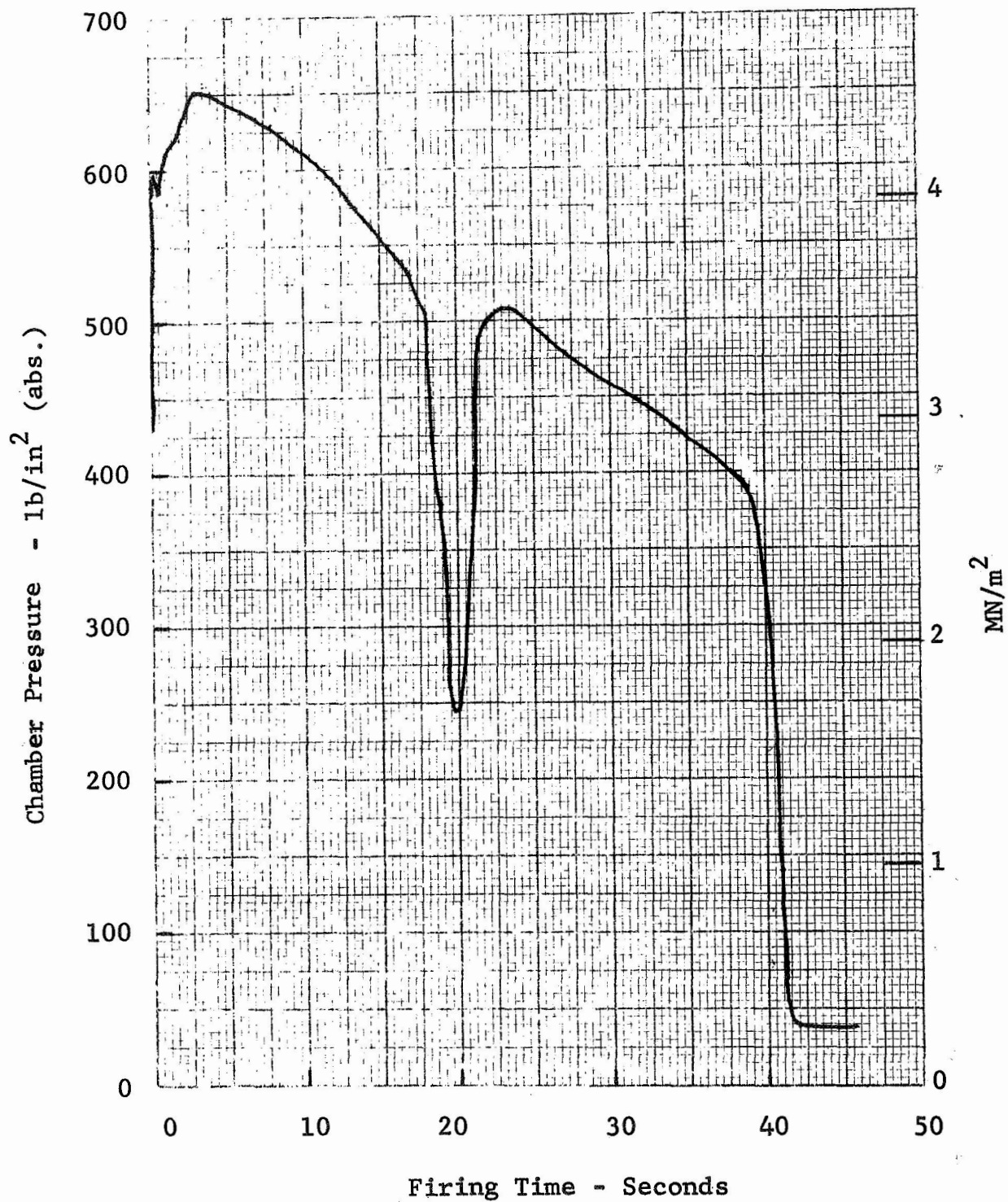
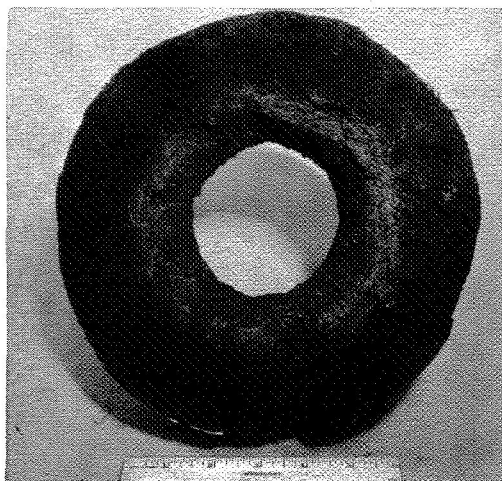
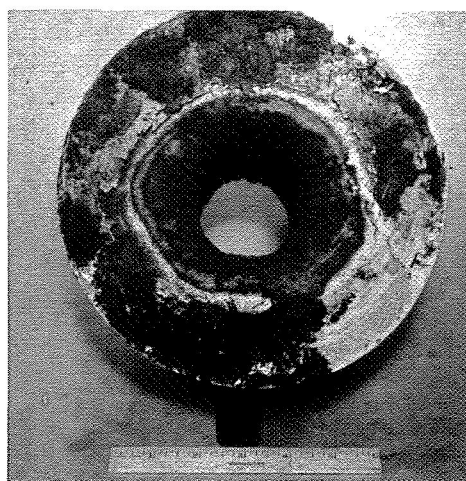


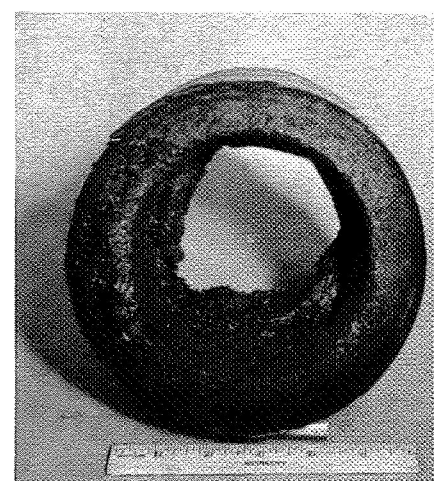
Fig. 23 VARIATION OF CHAMBER PRESSURE WITH TIME DURING FIRING OF NOZZLE 2.



Nozzle 1 Entrance



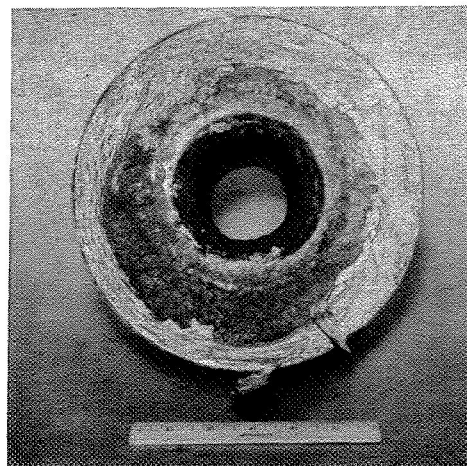
Nozzle 1 Throat



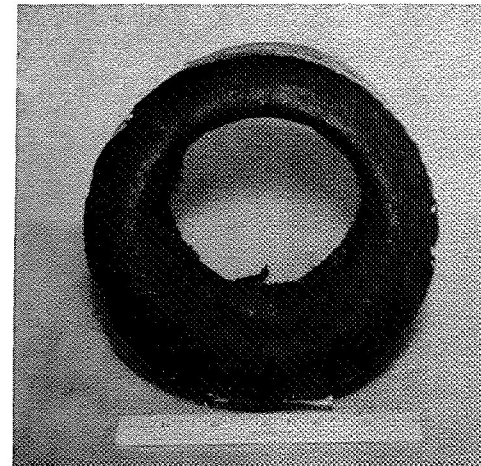
Nozzle 1 Exit



Nozzle 2 Entrance

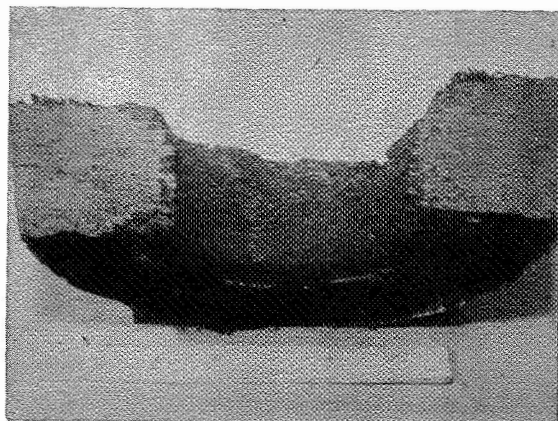


Nozzle 2 Throat

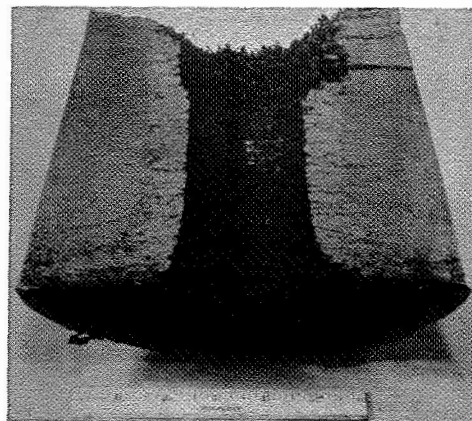


Nozzle 2 Exit

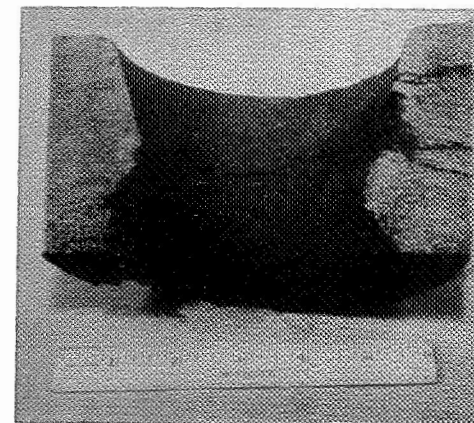
Fig. 24 NOZZLE INSERTS AFTER FIRING



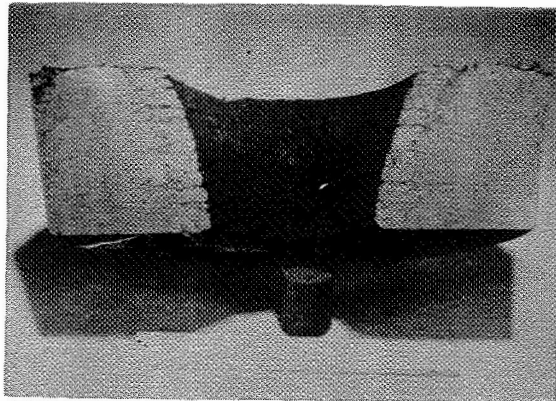
Nozzle 1 Entrance



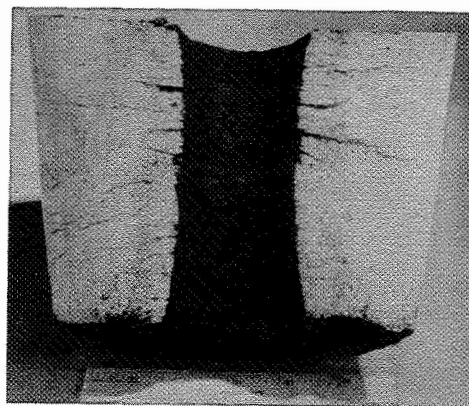
Nozzle 1 Throat



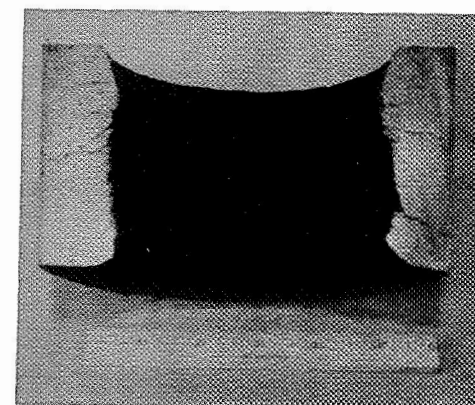
Nozzle 1 Exit



Nozzle 2 Entrance



Nozzle 2 Throat



Nozzle 2 Exit

Fig. 25 CROSS SECTIONS OF FIRED NOZZLES

Table XIV
DEPTH OF EROSION (MILS) IN ROCKET NOZZLE MATERIALS
FIRED UNDER SIMILAR CONDITIONS *
(CONVERTED TO 600 PSIA CHAMBER PRESSURE)

STATION** MATERIAL	ENTRANCE			THROAT						EXIT		
	12	11	10	9	8	7	6	5	4	3	2	1
1. LCCM 2626	77	35	108	38	54	64	59	21	13	--	--	--
2. IITRI 2	92	132	184	209	207	240	183	121	46	61	36	17
3. ARC 100	96	161	277	455	557	776	770	671	455	406	-12	23
4. MXC-12449	75	223	323	--	--	--	--	--	--	-15	-48	-40
5. IITRI 1	510	476	707	379	426	383	329	219	93	116	39	29
6. 4C1686	--	--	--	189	145	158	141	86	40	--	--	--
7. LCCM 4120	--	--	--	--	--	--	--	--	--	-12	-40	-50
8. MXC-113	--	--	--	--	--	--	--	--	--	124	11	6

Material Designations

1. LCCM 2626 Graphite filler, phenolic molding compound, pressed at 1000 lb/in², 150°C.
2. IITRI 2 Carbon filler, furfuryl ester resin, staple carbon fibers, pressed at 25 lb/in², cured at 150°C.
3. ARC 100 Molded carbon filler-resin.
4. MXC-12449 Carbon cloth, resin, vacuum-bag cured.
5. IITRI 1 Carbon filler, furfuryl ester resin, staple carbon fibers, pressed at 30 lb/in², cured at 150°C.
6. 4C1686 Carbon cloth, polyphenylene resin, unspecified filler, tape wrapped and cured at 250 lb/in², 177°C.
7. LCCM 4120 Graphite filler, phenolic casting resin, cast and cured at 15 lb/in², 77°C.
8. MXC-113 Carbon fiber paper, phenolic resin, tape wrapped and cured.

*Information from Aerojet Solid Propulsion Company.

**Locations of stations are shown in Figure 21.

under similar conditions. The IITRI no. 2 nozzle compared favorably with the other types, showing the second lowest erosion loss at most measuring stations. The body showing the lowest losses was a graphite-phenolic resin combination which was molded at 1000 lb/in² (6.89 MN/m²). This high fabrication pressure produced a greater density, but it would require heavy presses to mold large pieces. In contrast, the highest pressure used for an IITRI insert was 36 lb/in² (0.25 MN/m²).

VI. CONCLUSIONS

As a result of this effort to develop improved carbonaceous composite formulations for use in high-performance, low-cost rocket nozzles, the following conclusions may be drawn:

1. The optimum composition for the moldable material is

Petroleum coke	52%
Carbon black	5
Carbon fibers	8
Binder	<u>35</u>
	100

2. For pressing small samples or large billets it was not necessary to use high pressures. Values between 10 and 60 lb/in² (0.069 and 0.41 MN/m²) were adequate when the material was partially cured under load.

3. Density and thermal shock in these samples were found to be inversely related. The specimens with the best records for resisting cracking and spalling in a plasma torch test were those with low densities - below 1.35 g/cc.

4. The incorporation in the mixture of a few percent of staple carbon fibers was very useful in resisting cracking and the propagation of existing cracks. Coating of the fibers with binder before use aided fiber-matrix bonding, but this expensive process may be dispensable if the improved fabrication procedures described in section IV of this report are followed.

5. A study of the economic factors involved in producing bodies of CFA material showed that the very high cost of using small quantities of materials and laboratory techniques could be substantially reduced. Taking advantage of commercial purchasing and processing economies and using a low-cost organic fiber for reinforcement, the cost per pound of unfinished nozzle could be reduced by a factor of 10. The best value of \$2.39/lb (\$5.29/kg) was close to the original goal of \$2.00/lb (\$4.41/kg).

6. Firing tests on two subscale nozzles showed that the entrance and throat inserts should be pressed to a density of 1.50 to 1.60 g/cc for a satisfactory balance between thermal shock and erosion resistance. The exit insert may be pressed to lower density because of the less severe conditions in that region of the nozzle.

7. A comparison between the nozzles prepared for this program and several other types containing various forms of carbon showed that the higher density IITRI version (no. 2) was second only to a similar model fabricated under much greater pressure. The advantages of the low-pressure process (10 to 60 lb/in², 0.069 to 0.41 MN/m²) are the simplicity and economy of the dies and press and the speed and ease of fabrication.

VII. RECOMMENDATIONS

Some additional development work is recommended to improve the properties and lower the cost of the CFA system. The following paragraphs list some areas in which important progress may be made.

1. The petroleum coke filler used in this work was specially ground to pass a 325-mesh screen. The less expensive -200 mesh commercial product will probably do as well if the particle size groups are properly balanced.

2. Rayon and polyacrylonitrile yarn deserve more intensive evaluation than time permitted in this program. The economic aspect is a very strong incentive to change, and the higher quality of the large volume organic yarns is also important.

3. Staple fibers which have not been precoated with binder should be thoroughly tested to determine whether they can serve their purpose without a coating. Since coating is a relatively expensive step, its elimination would be worthwhile.

4. The low-pressure compaction process with external heating, which was used at the end of the program, should be instituted as standard practice, especially for large samples.

5. The use of carbide powder as an additive to the CFA material adjacent to the hot surfaces of inlet and entrance inserts should be investigated as a solution to the erosion of these sections by particulate matter in the propellant gases.

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